

Mass Spectrometry and Gas Chromatography on Entry Probes: Past, Present, and Future

IPPW #3

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Unique Features of Entry Probe Experiments

- Extreme conditions during atmospheric entry
- Time constraints due to the short duration of the experiment
- Rapid environmental changes in temperature and pressure during descent
- Resource limitations, i.e. mass, power, size and bandwidth
- Instrument design driven by conditions (pressure, temperature, composition) which vary widely from planet to plane



Entry Probe Mass Spectrometers

- Mass spectrometer
- Sample inlet and conditioning system (plumbing)
- Pumping system
- Electronics



Past Missions

- PAET: June 1971
- Venera 9 and 10: October 1975
- Viking 1 and 2: July and September 1976
- Pioneer Venus Multiprobe: December 1978
- Venera 13 and 14: March 1982
- Vega 1 and 2: June 1985
- Galileo Probe: December 1995
- Cassini-Huygens Probe: January 2005
- Venera 11 and 12: December 1978



Existing Spaceflight Mass Spectrometer Technology



Engineering Model of Huygens Probe GCMS

1000's vacuum seals

Dozens of custom parts from multiple vendors:
• micro-valves
• electron multipliers
• pumps
• GC columns & other plumbing

Custom hand-made assemblies
• quadrupole rods
• pressurized housing

1000's of COTS parts
• electronic components
• fittings, bolts, nuts, switches, etc.

Desired Characteristics for Future Entry Probe Mass Spectrometers

Reduced power, mass and volume, which would allow:

- mass spectrometers on space/power constrained missions
- parallel measurements (e.g., simultaneous direct inlet, GC)
- multiple entry probes
- redundancy

Improved Performance:

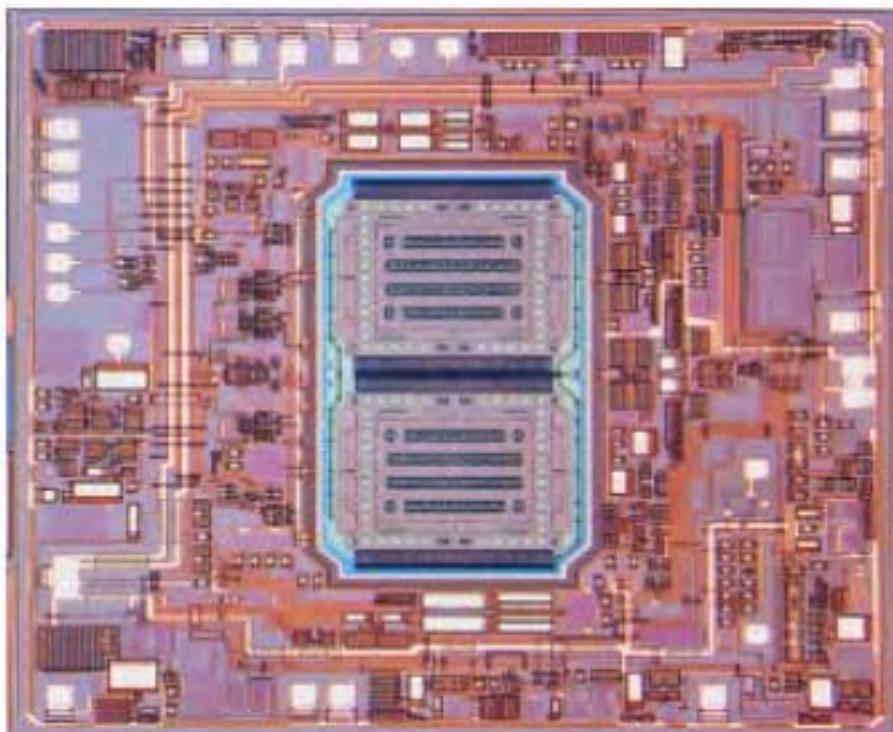
- reduced background chemical noise
- greater mass range, without sacrificing resolution

Increased Instrument Integration:

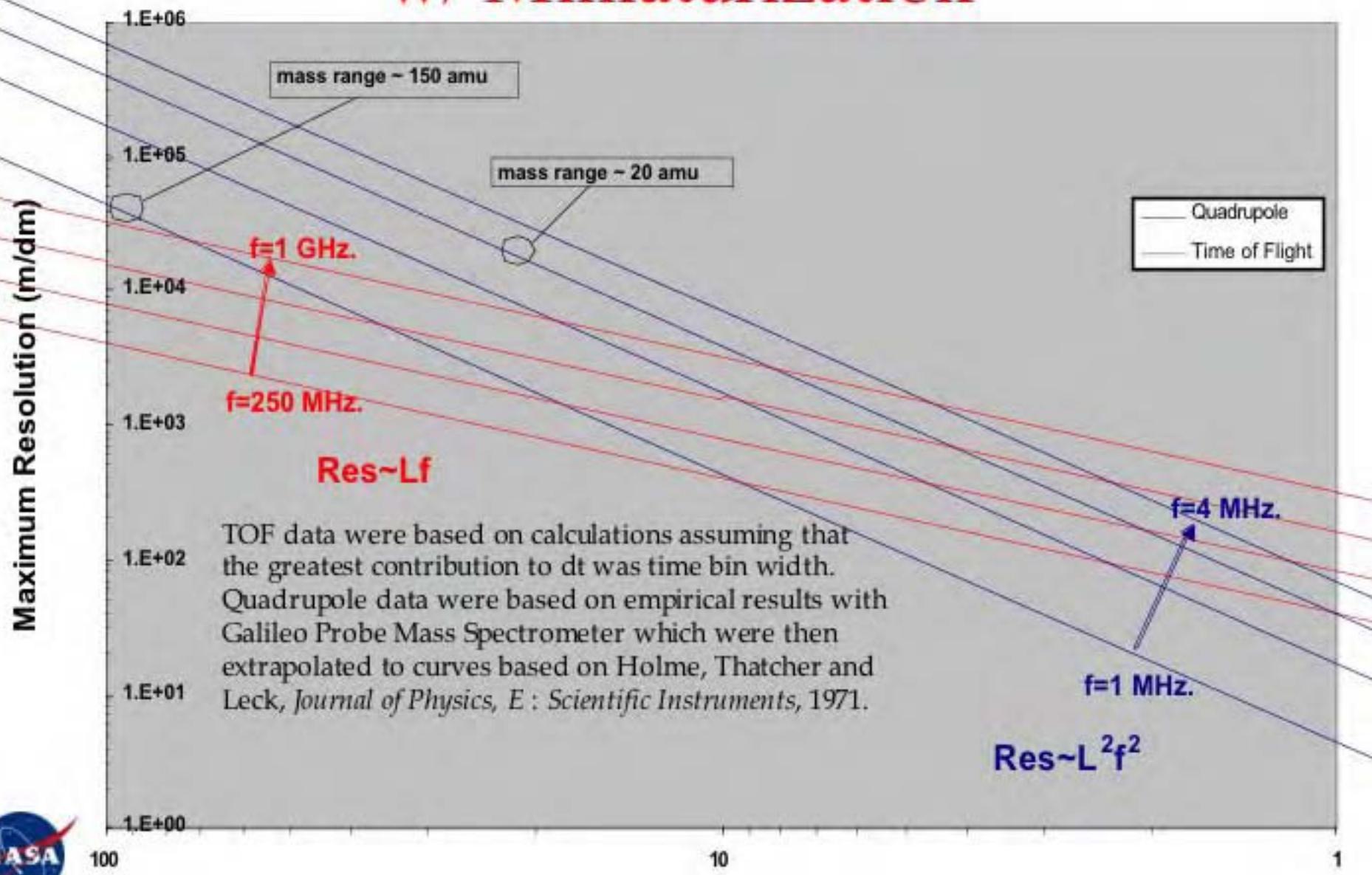
- reduction in the number of separately developed components and sub-systems
- integration of instrument and entry heat shields
- reduced integration complexity and cost



Illustration of System Integration through MEMS

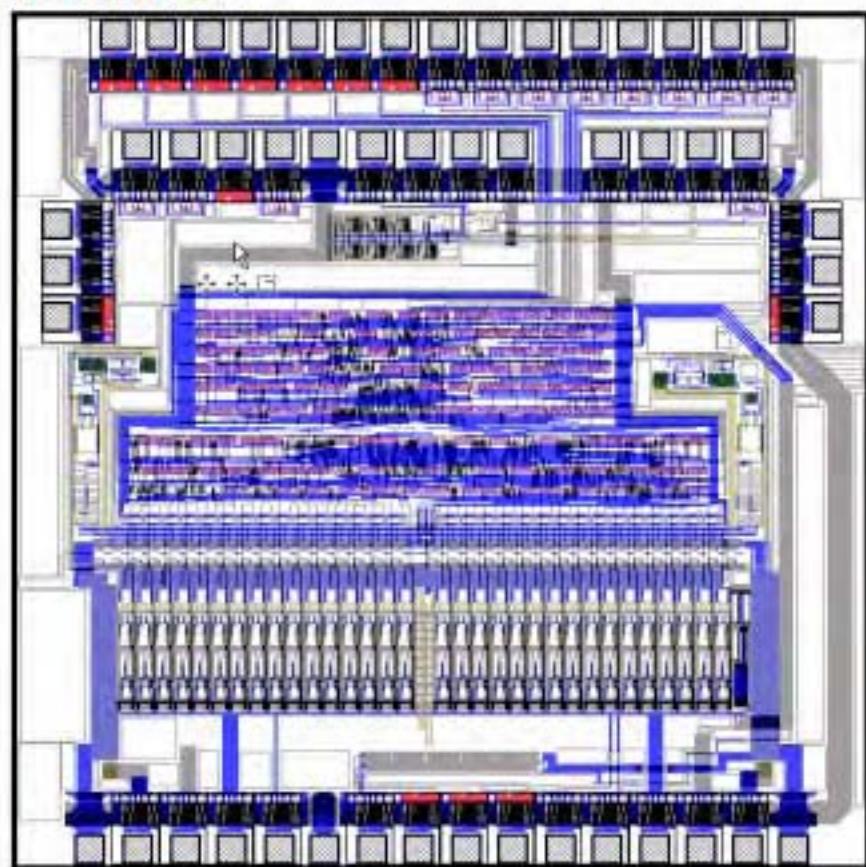


Mass Filter Performance Trade-offs w/ Miniaturization



2 GHz. Multi-event Time-of-Flight to Digital ASIC

- High speed 2 GHz. time-to-digital converter chip designed at GSFC and fabricated through MOSIS
- Reduces time bin widths and maintains resolution at smaller length scales
- Inherent tradeoff between mass resolution and mass range is avoided
- Low voltage integrated circuit has modest power requirements

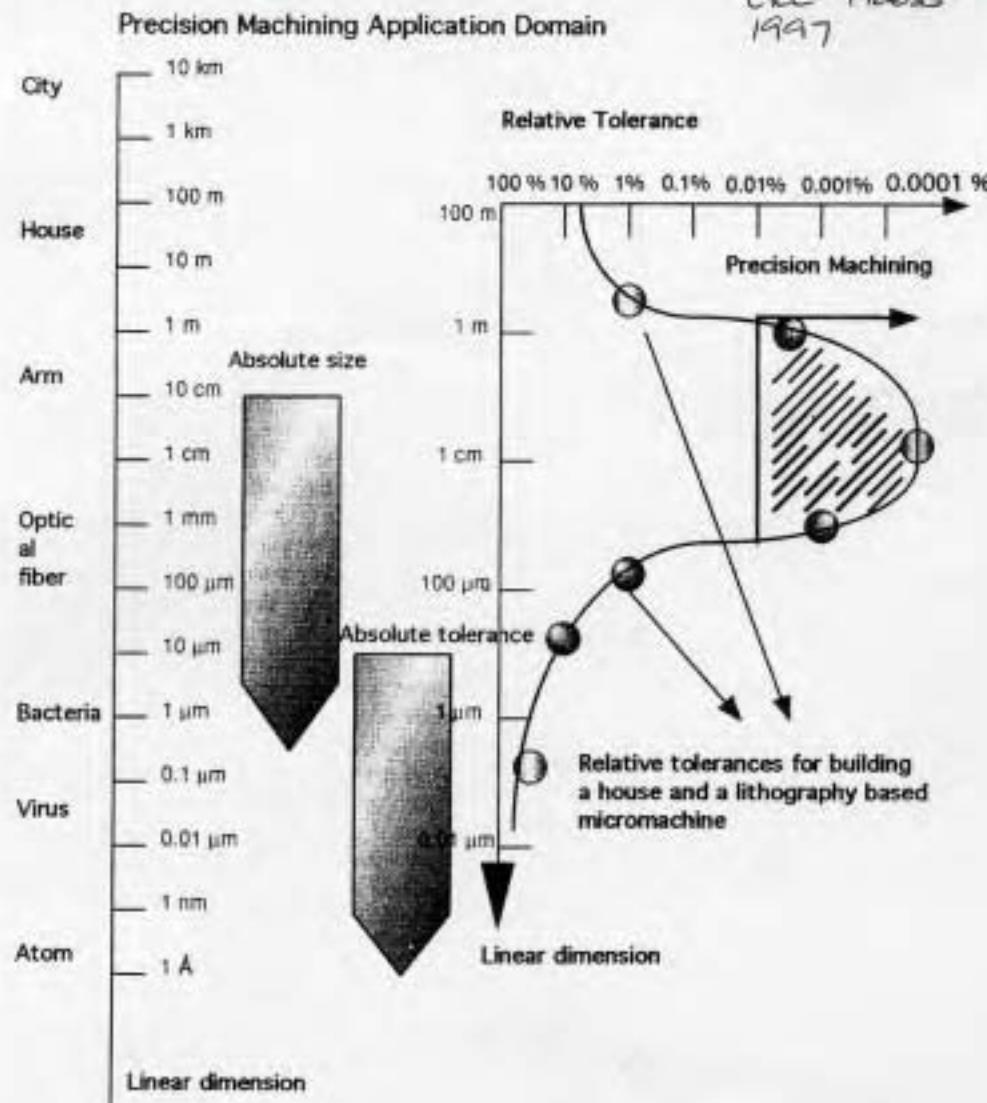


Design & Layout by Jeff Dumonthier, Steve Feng, Dave Sheppard, Miles Smith, George Winkert, NASA/GSFC.



Trade-offs With Miniaturization

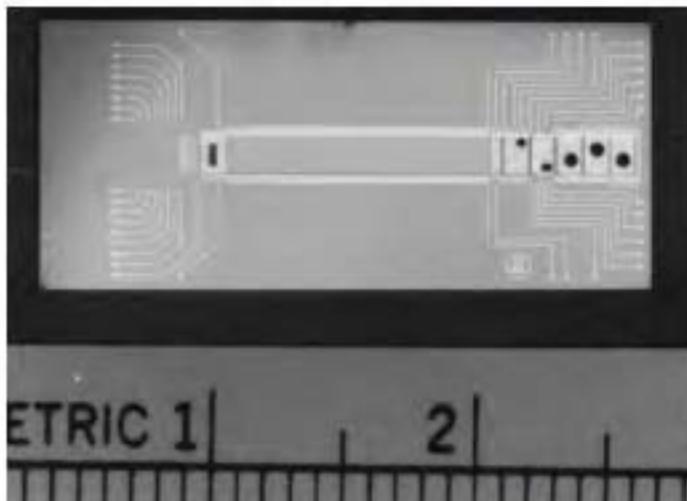
Fundamentals of Microfabrication
MARC MADOU
CRC PRESS
1997



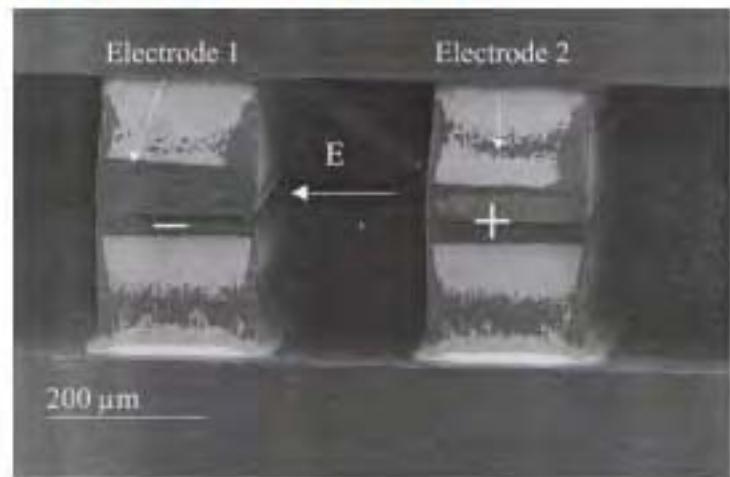
Micro-fabricated Mass Filters:



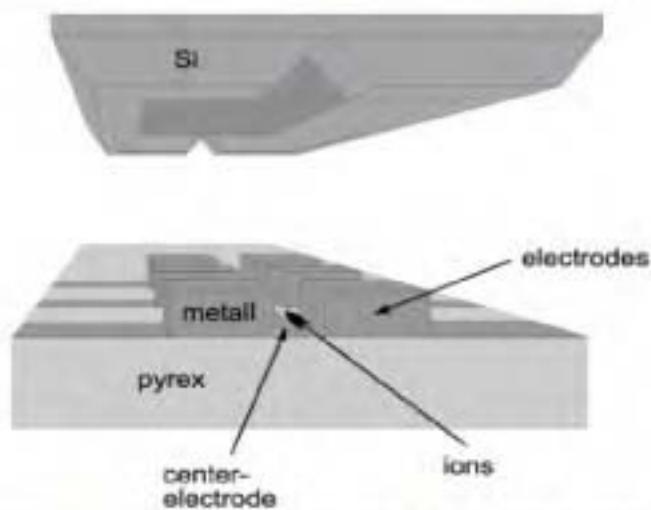
LIGA Quadrupole Array, Eyre,
George et al, JPL, (2000).



NASA
Micro-fabricated ion lens and Wien filter,
Freidhoff, et. al., *J. Vacuum Sci. Tech.*, (1999).

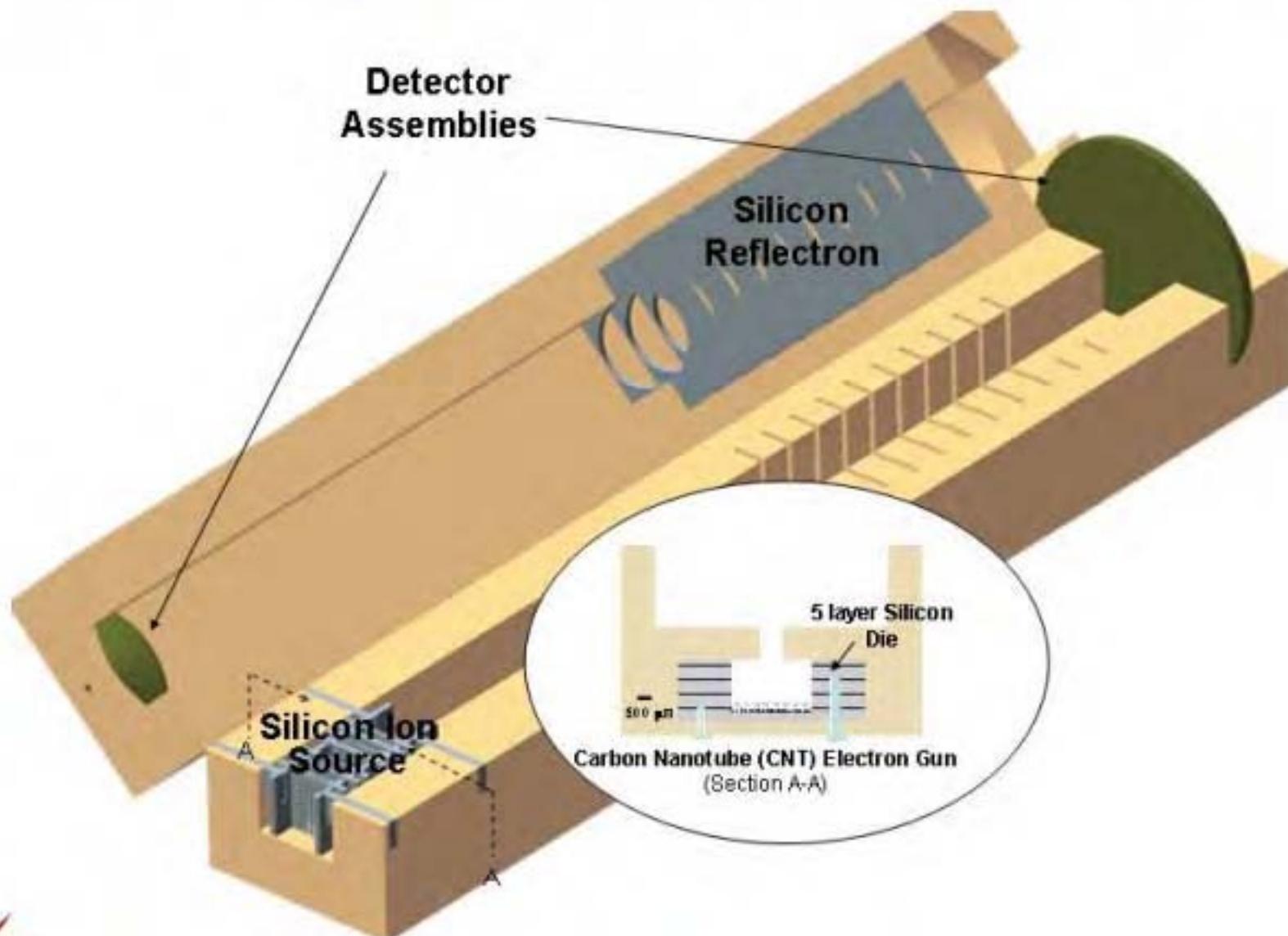


Micro-fabricated Wien filter, Sillon, Baptist,
et. al., *Sensors and Actuators B* (2002).



Traveling wave time-of-flight filter concept
Sillon , et. al, *App. Phys A*, (1998).

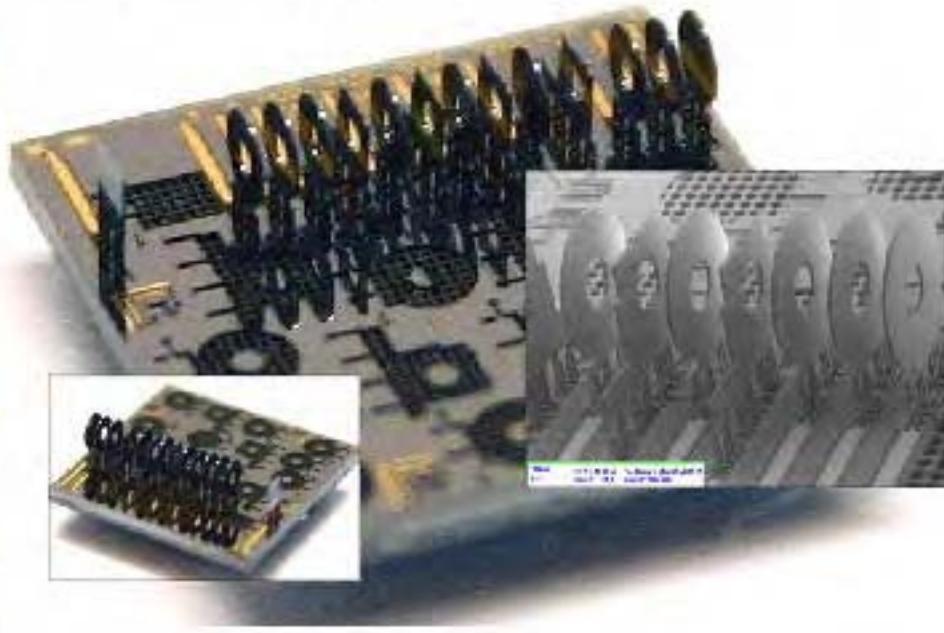
Time-of-flight MEMS Mass Spectrometer



Ion Optics for MEMS Time-of-flight Mass Spectrometer

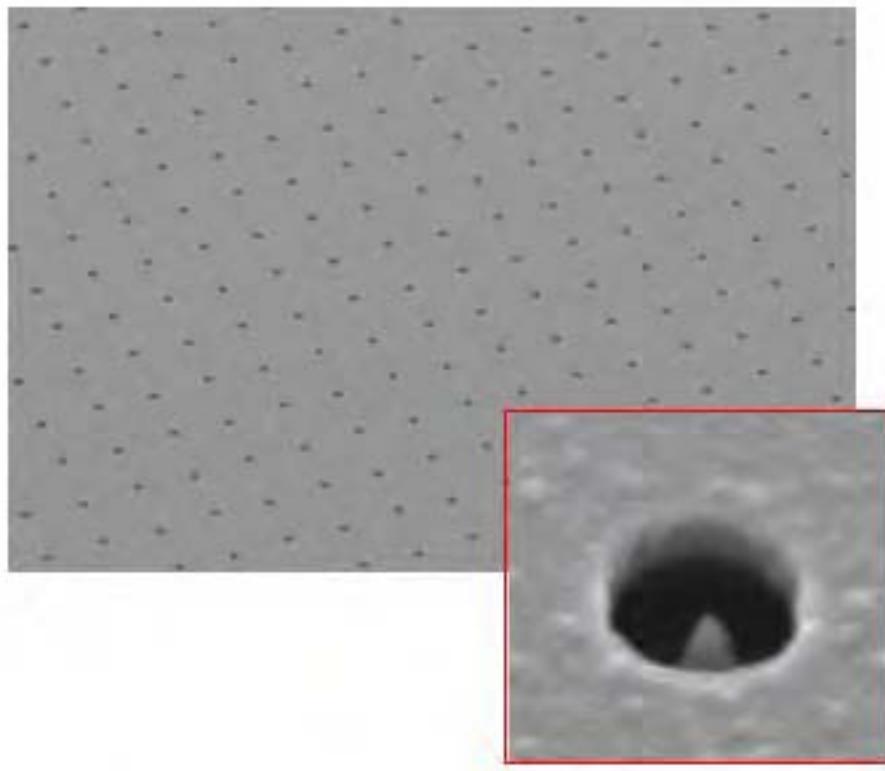


Machined silicon ion optics fabricated and assembled in-house.

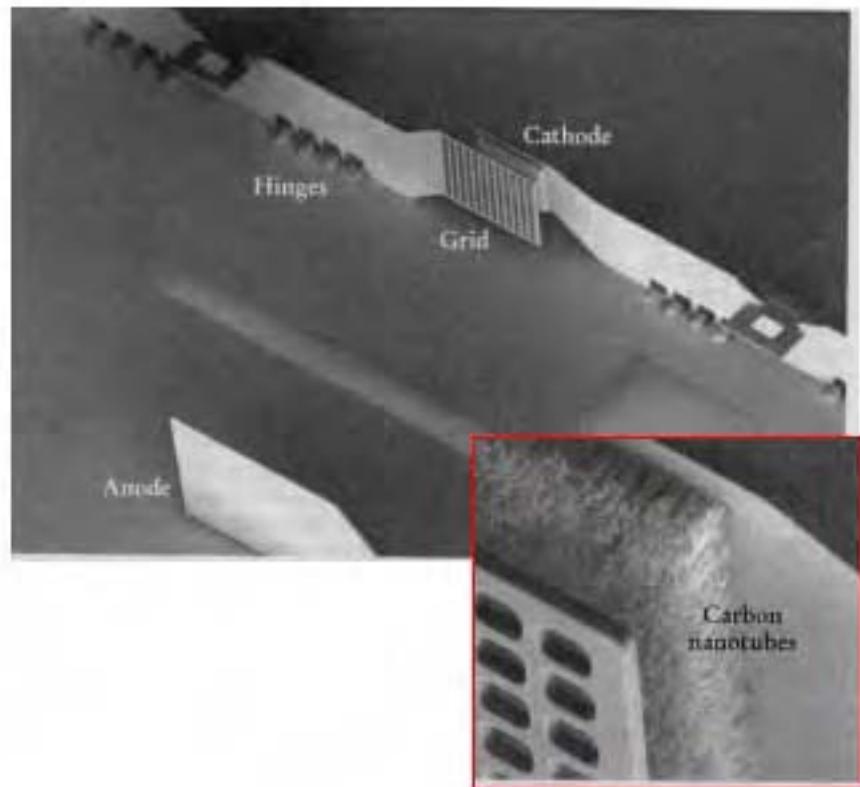


GSFC is working with Zyvex Corporation to develop a monolithic (all silicon) ion source assembly based on the technology used to develop this miniature SEM column (above).

Micro-fabricated Electron Sources

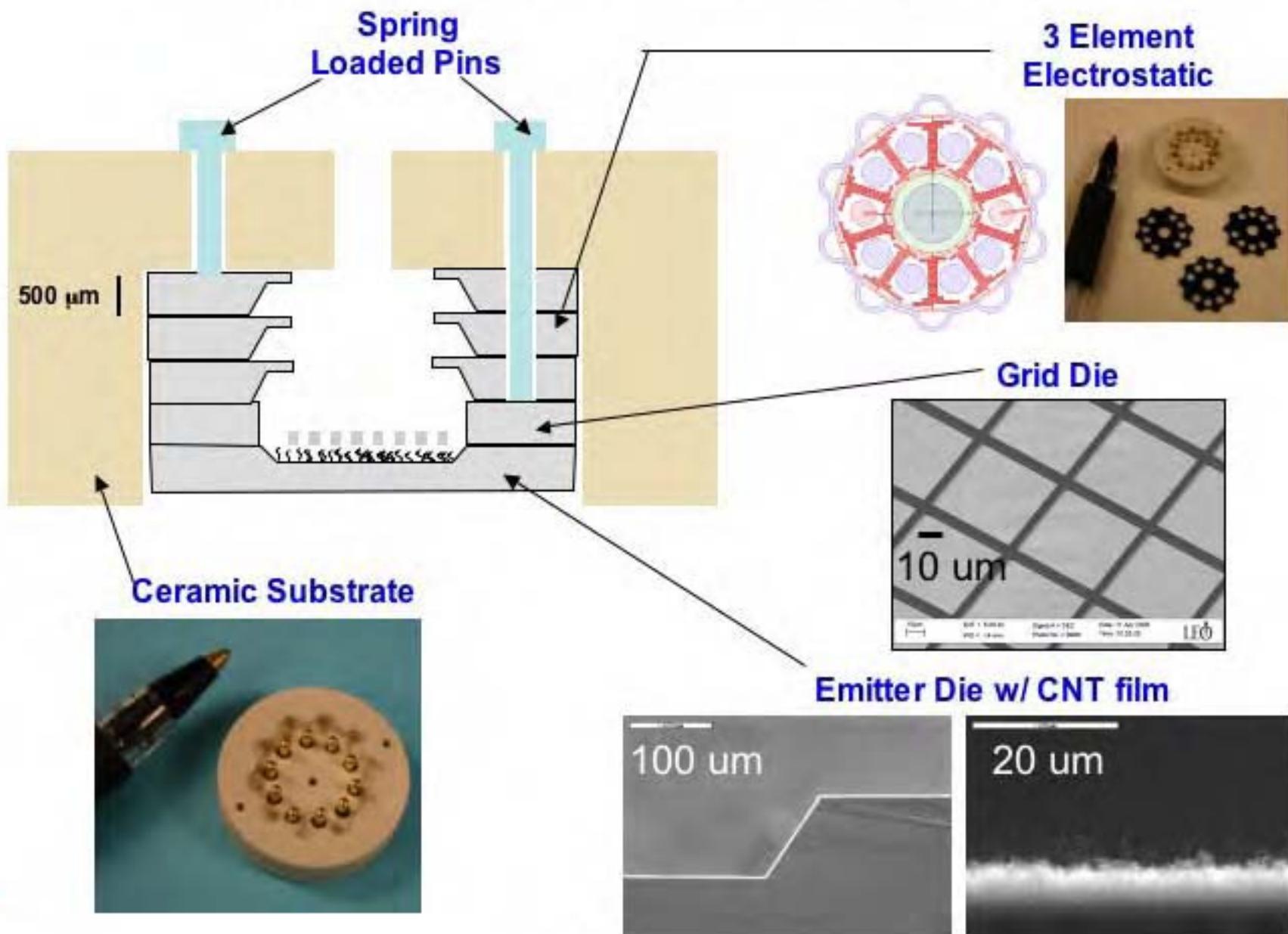


Molybdenum Spindt
Emitters, SRI
International.

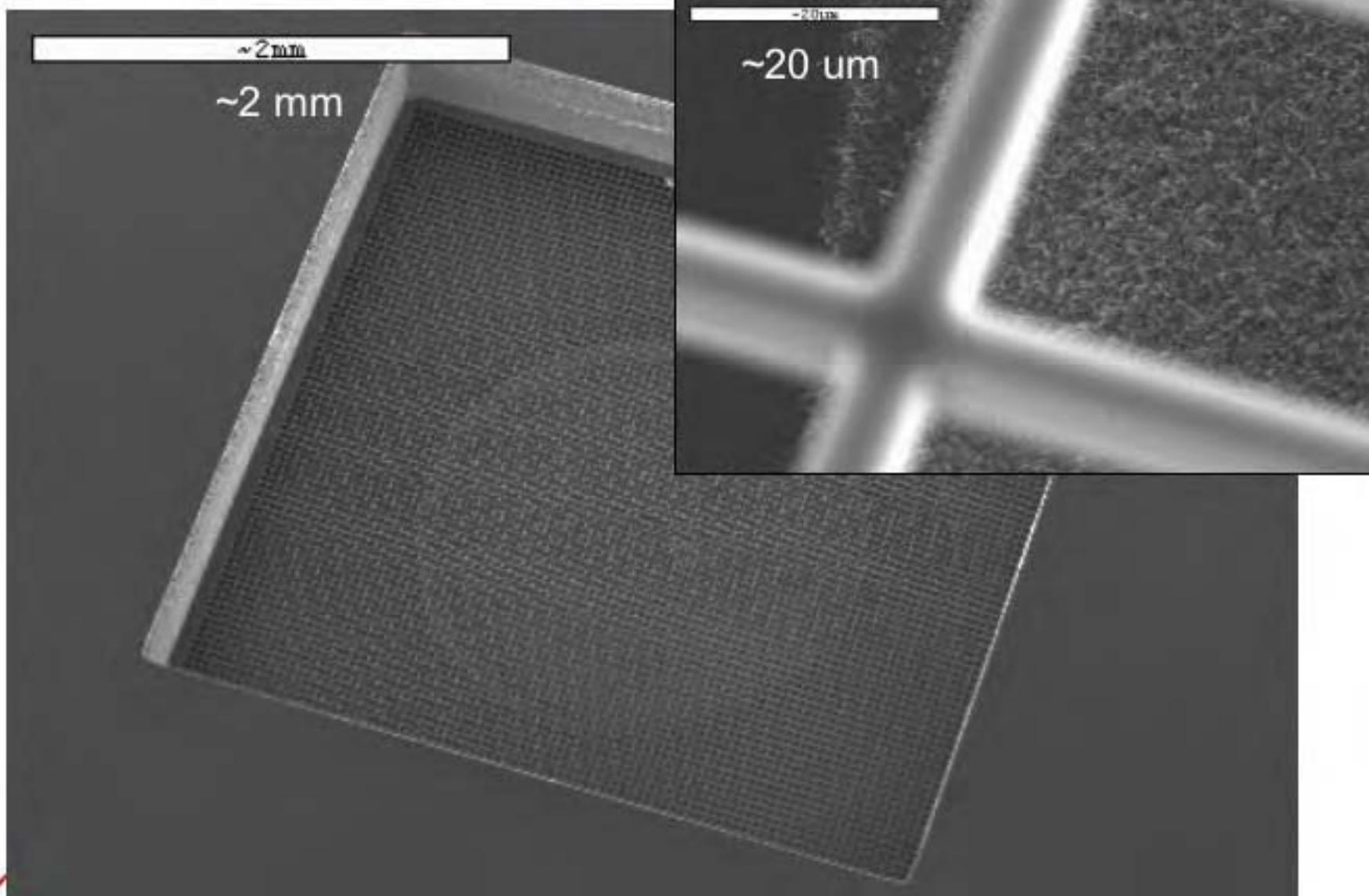


Carbon Nanotube Triode,
Bowers et. al, Applied
Physics Letters, (2000).

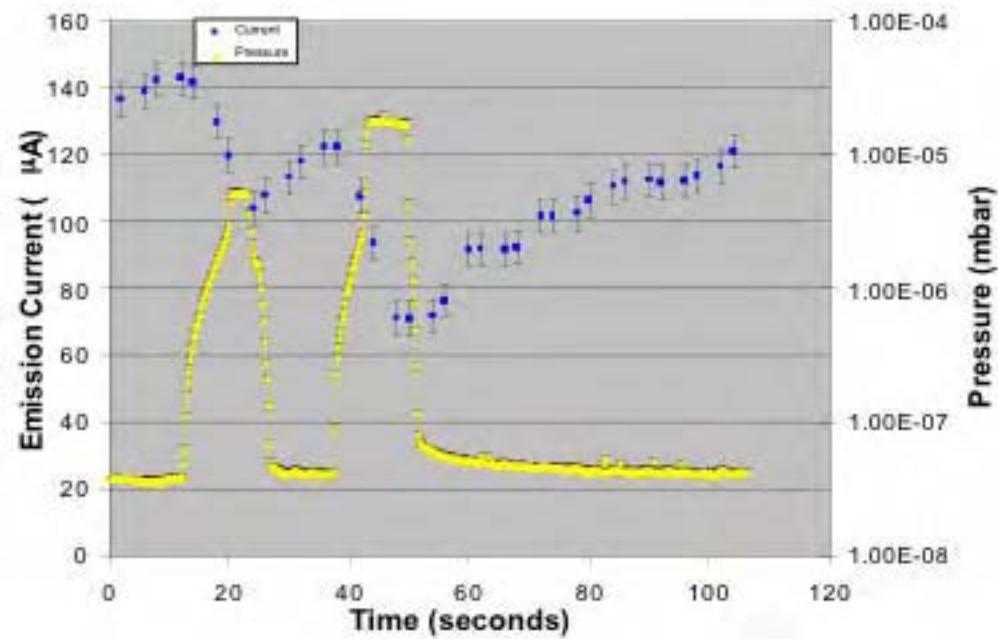
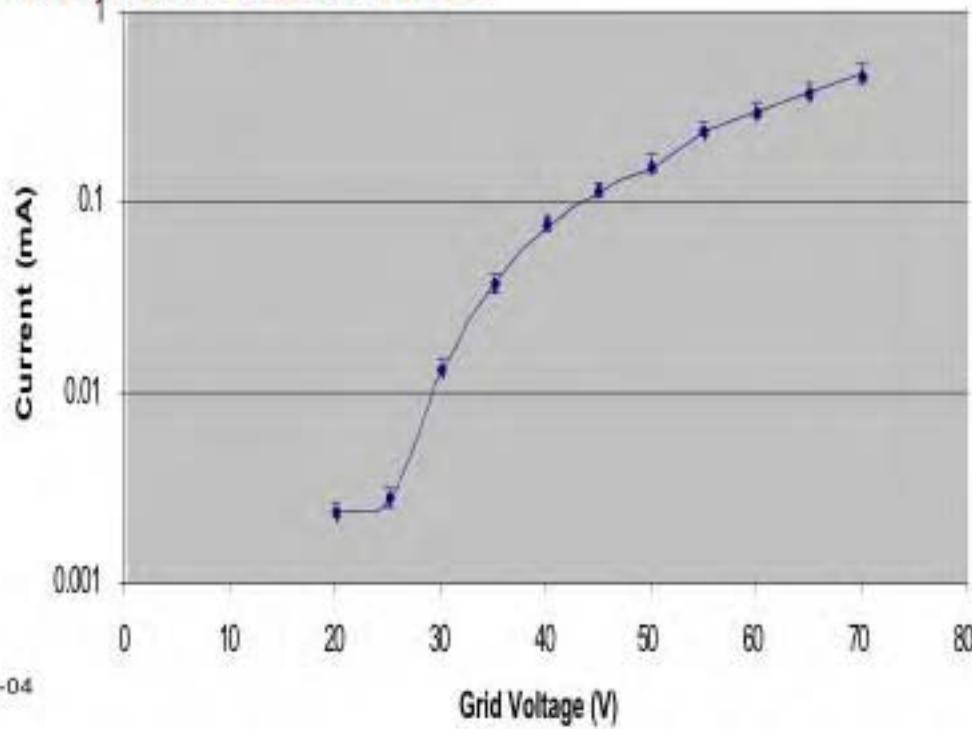
Carbon Nanotube Electron Gun



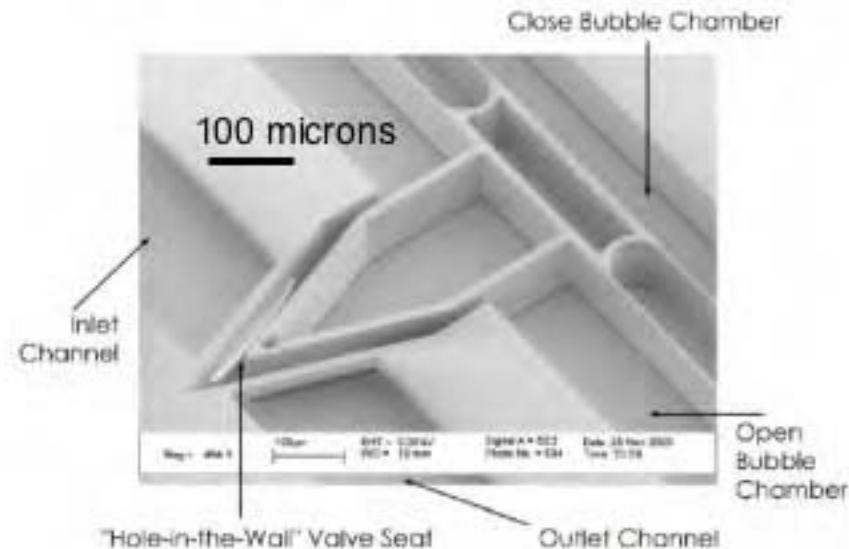
Carbon Nanotube Electron Emitter for Miniature Mass Spectrometer



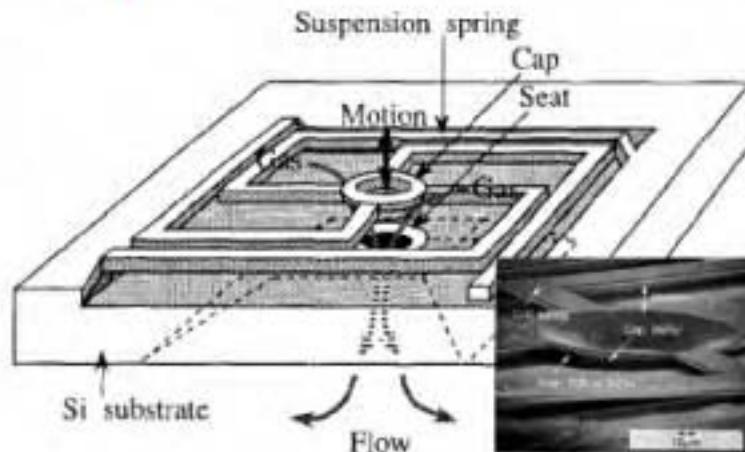
Emission Characteristics



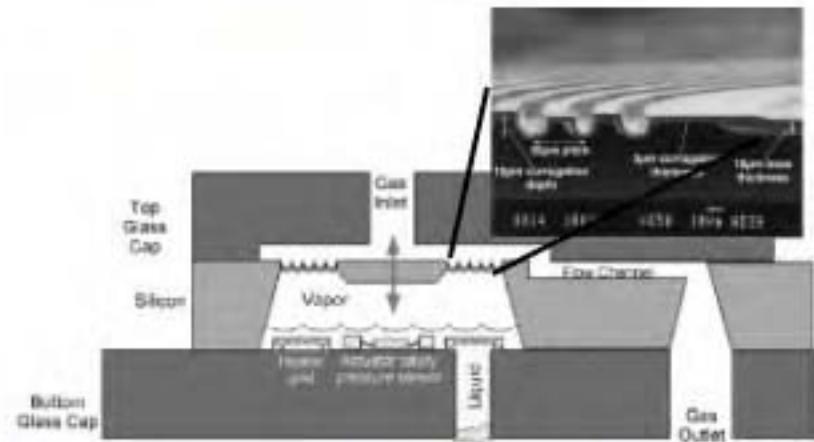
Instrumentation-quality Micro-valves



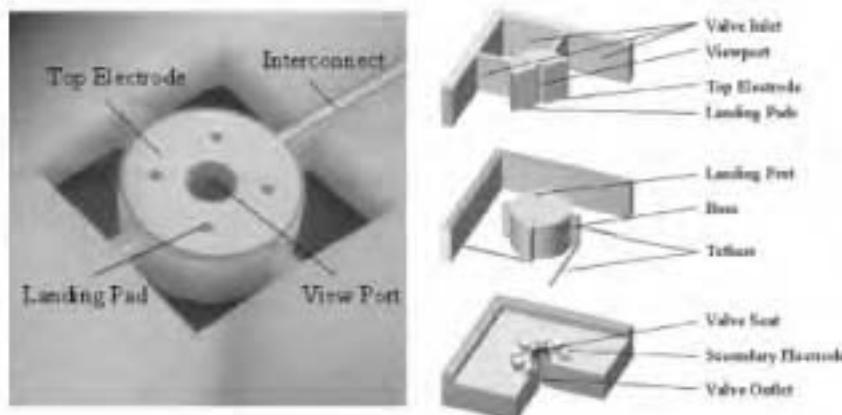
Gate Valve, Frank & Pisano, UC Berkeley



Magnetically Actuated Valve, Hirano, Nakano et. al., NTT Integrated Information & Energy Systems Laboratories, Japan.



Thermo-pneumatically Actuated Valve,
Rich & Wise, U. Michigan



Electrostatically Latched Normally Closed Valve, Yang, Schmidt et. al., MIT



MEMS Pumps

Summary of MEMS Pump Performance

Laser, D. J. and J. G. Santiago (2004), "A Review of Micropumps,"
Journal of Micromechanics and Microengineering **vol.14, no.6:** R35-R64.



MEMS Valves and Pumps for Planetary Probes: Factors to Consider

Performance specifications:

- valves: leak rate? (typ. reported 10^{-3} sccm)
- pumps: pumping speed (see previous chart) and maximum differential pressure (typ. reported 10's of kPa.)
- may have to sacrifice some specs and make up in other areas (e.g., increased sensitivity w/miniatrization allows lower gas sampling rate, allowing "leakier" valves and lower pumping speeds)

Materials: vacuum compatible & bakeable?

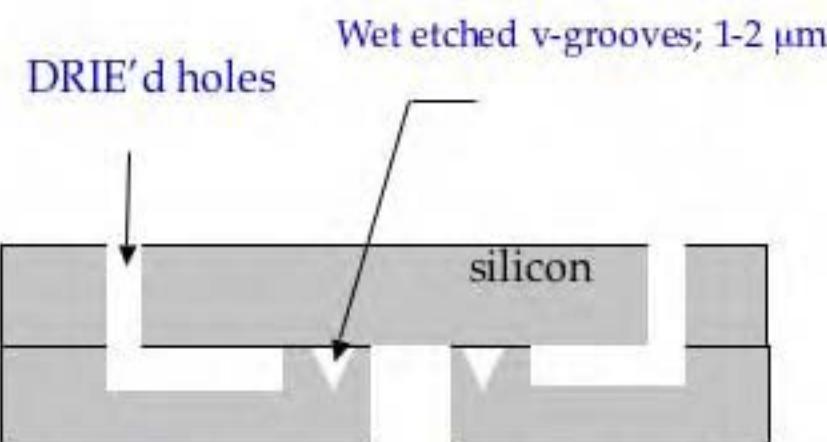
Packaging and Integration: is it possible to integrate the components with existing systems?

Are there better ways of doing things, unique to this application?

- e.g., sputter/ion pumps (which are not getting much attention) may be a good way to go since the total volume of gas needed to be pumped is low...
- one-shot valves for sampling instead of fully operable valves? (see next slide)



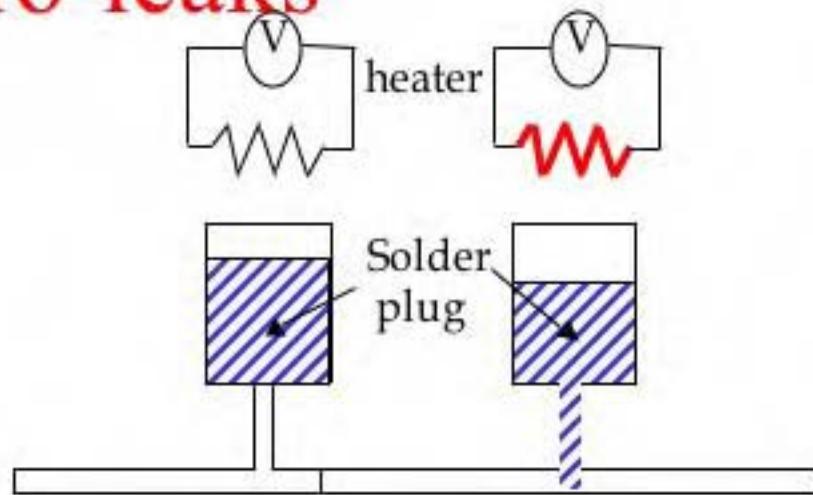
Silicon Micro-leaks



Fabrication process for silicon micro-leaks.



Assembled micro-leak die frit bonded into mini conflat flange.



OPEN
CLOSED
One-shot valve concept for sealing
micro-leak channels.

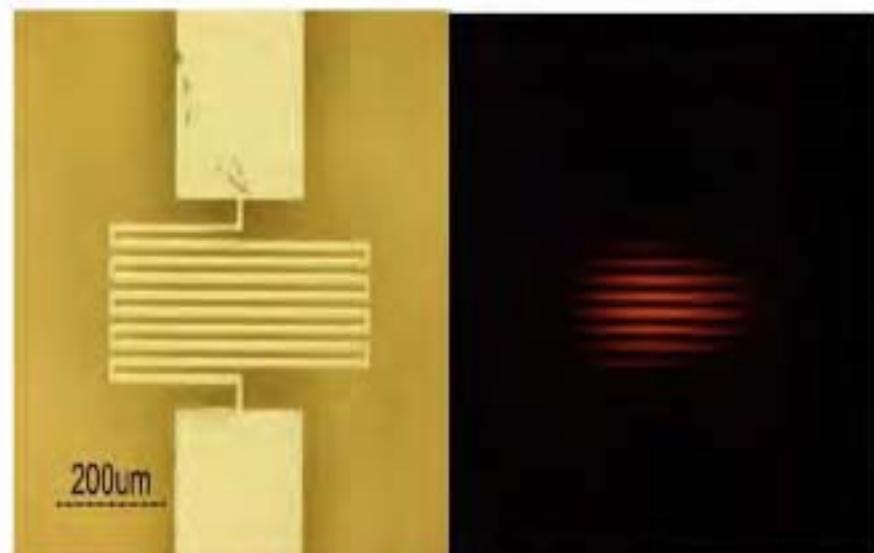
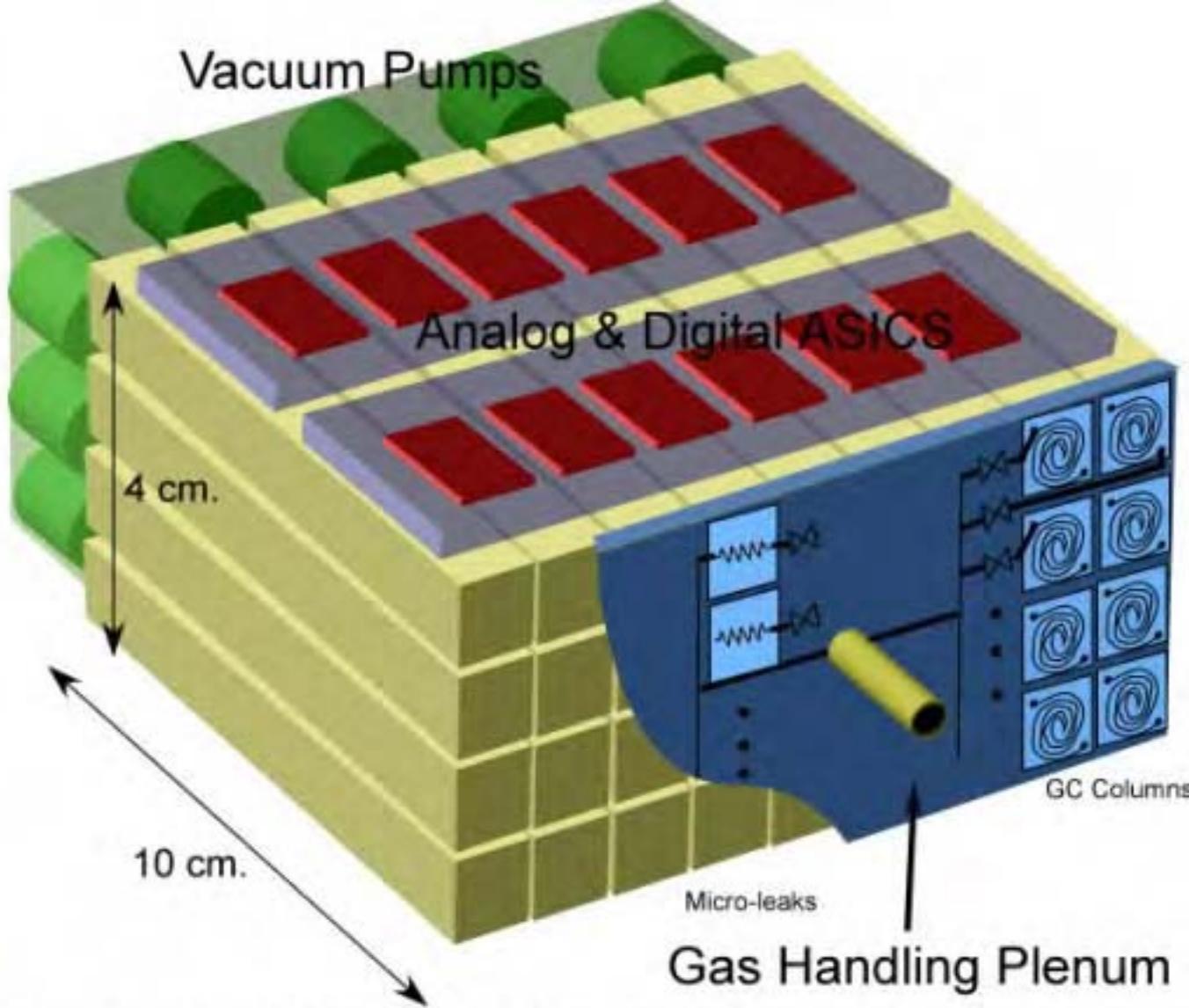


Figure 9.1 - a) Heater before test; b) Heater at 1.5 W
Fabricated heater for one-shot actuation.



- Mass Spectrometer redundancy allows for a safety margin with respect to failure, and allows simultaneous parallel measurements. Here, separate time-of-flight analyzers are shown sampling from multiple GC columns and direct atmospheric leaks (micro-leaks)

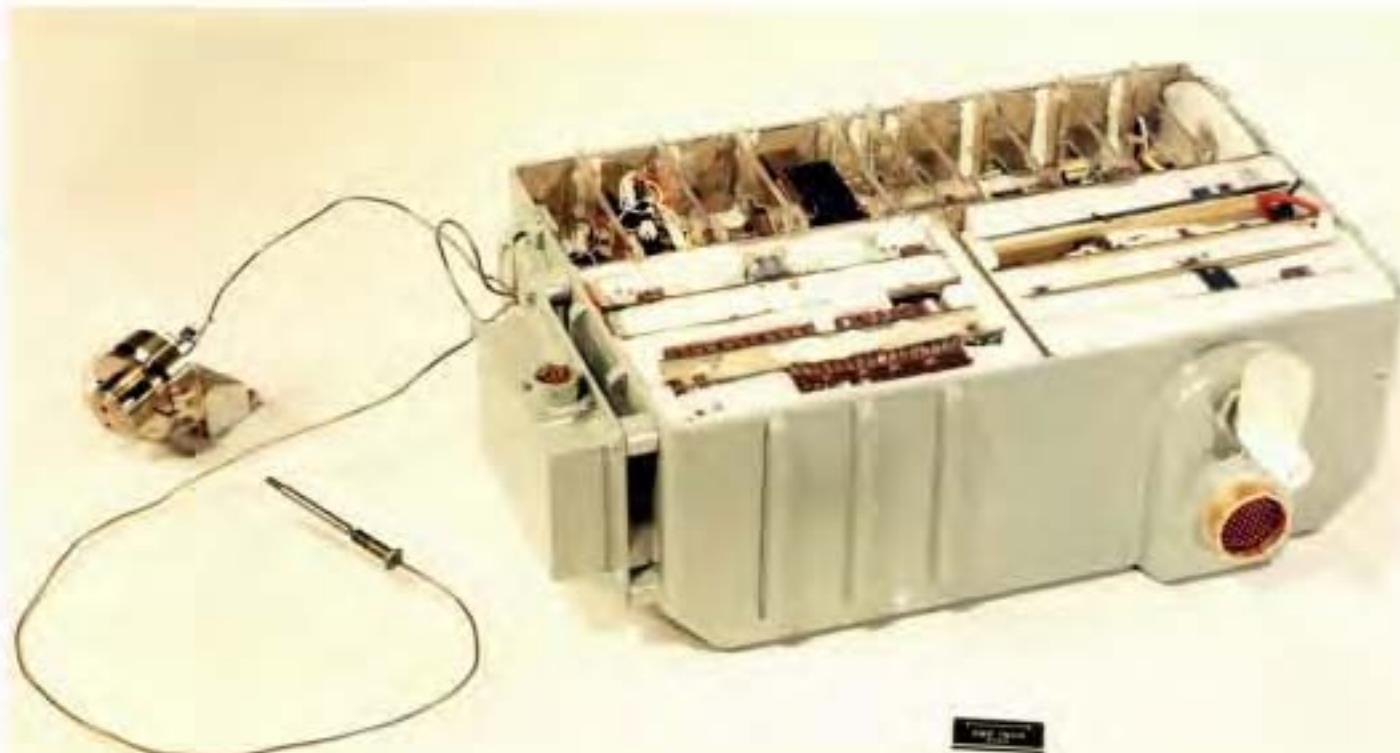
Supplemental Material



Planetary Atmospheric Entry Test (PAET)

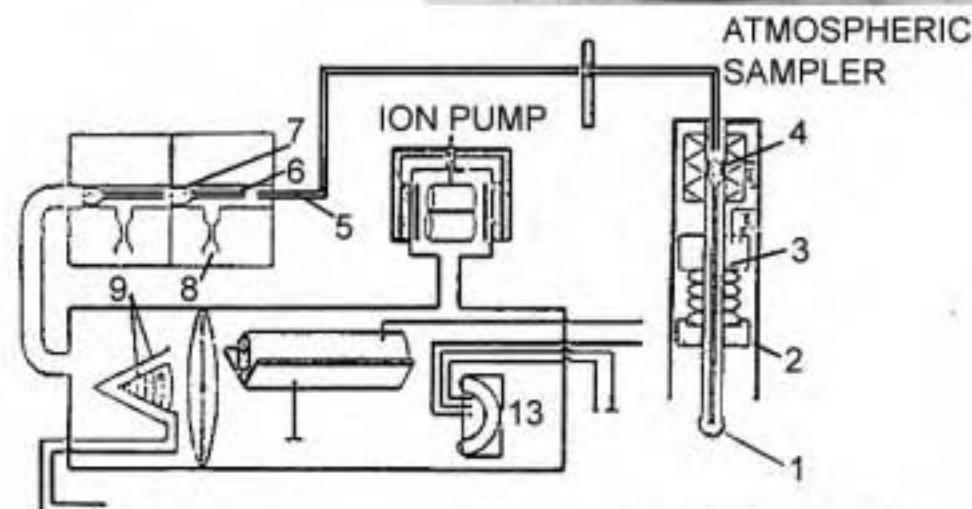
- Launch: June 20, 1971
- First entry probe mass spectrometer
- Quadrupole analyzer, 2 electron impact ion sources, secondary electron multiplier
- Mass: 1.6 kg
- Rod length:
15.24 cm
- Mass Range:
10-90 u

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Soviet Venera-9 and 10 Missions to Venus

- Launch: Jun. 8, 1975 (V-9); Jun. 14, 1975 (V-10)
- Entry: Oct. 22, 1975 (V-9); Oct. 25, 1975 (V-10)
- Lander mass: 660 kg (without aeroshell)
- First mass spectrometers (RF) used in an interplanetary mission
- Monopole analyzer, electron impact ion source, secondary electron multipliers
- Operated from 50 to 30 km
- Mass: 10 kg
- Average Power: 35 W
- Mass Range: 10-60 u



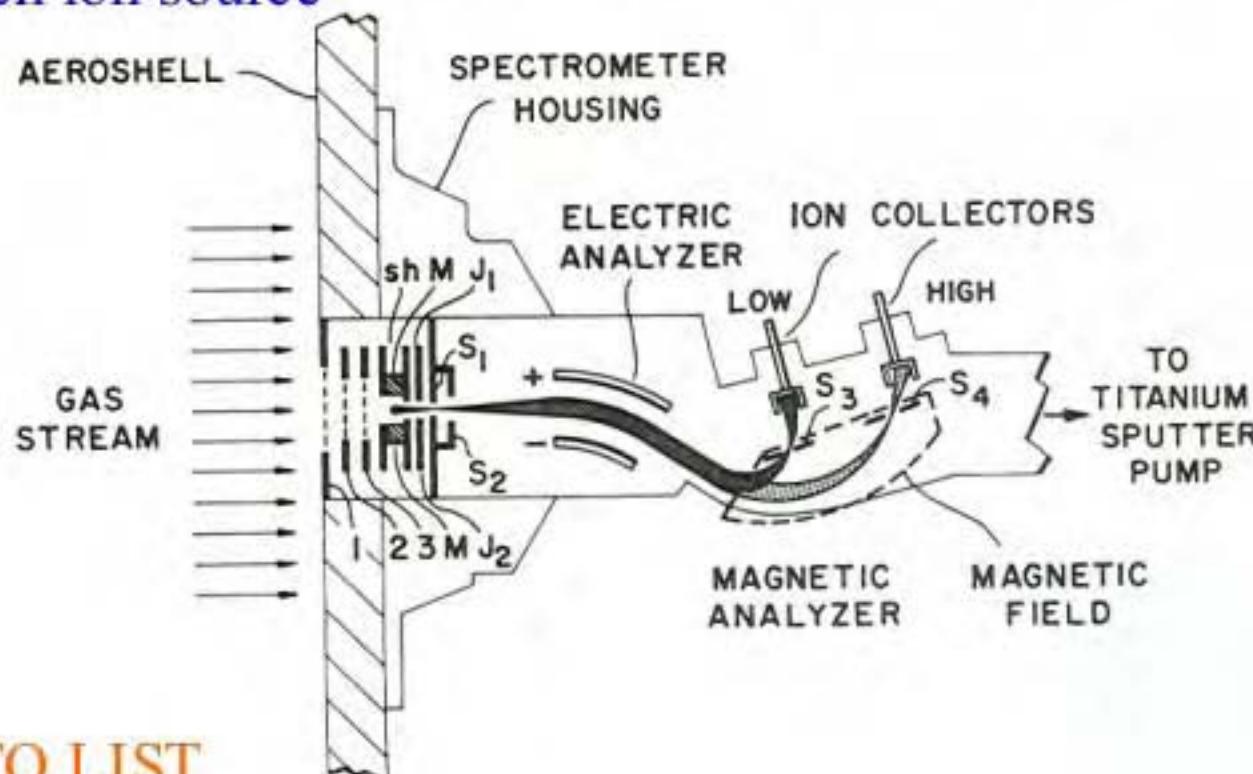
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Ref: Surkov, Yu A., 1977, Venus by Venera 9 and 10 automatic interplanetary stations, Proc. Lunar Sci. Conf. Volume 8, 2665-2689.

Viking 1 and 2 Neutral Mass Spectrometer (NMS)

- Launch: August 20, 1975 (Viking 1), September 9, 1975 (Viking 2)
- Entry and landing: July 20, 1976 (Viking 1), September 3, 1976 (Viking 2)
- Double-focusing (electrostatic and magnetic) mass spectrometer
- Electron-impact open ion source
- Mass: 6.2 kg
- Average Power:
13.3 W
- Mass Range:
1- 49 u



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Pioneer Venus Multiprobe

- Launched August 8, 1978
- Consisted of a large probe, 3 small probes, and the instrumented bus

- Large probe released November 16, 1978
- Small probes released November 20, 1978
- Atmospheric entry of probes and bus on December 9, 1978

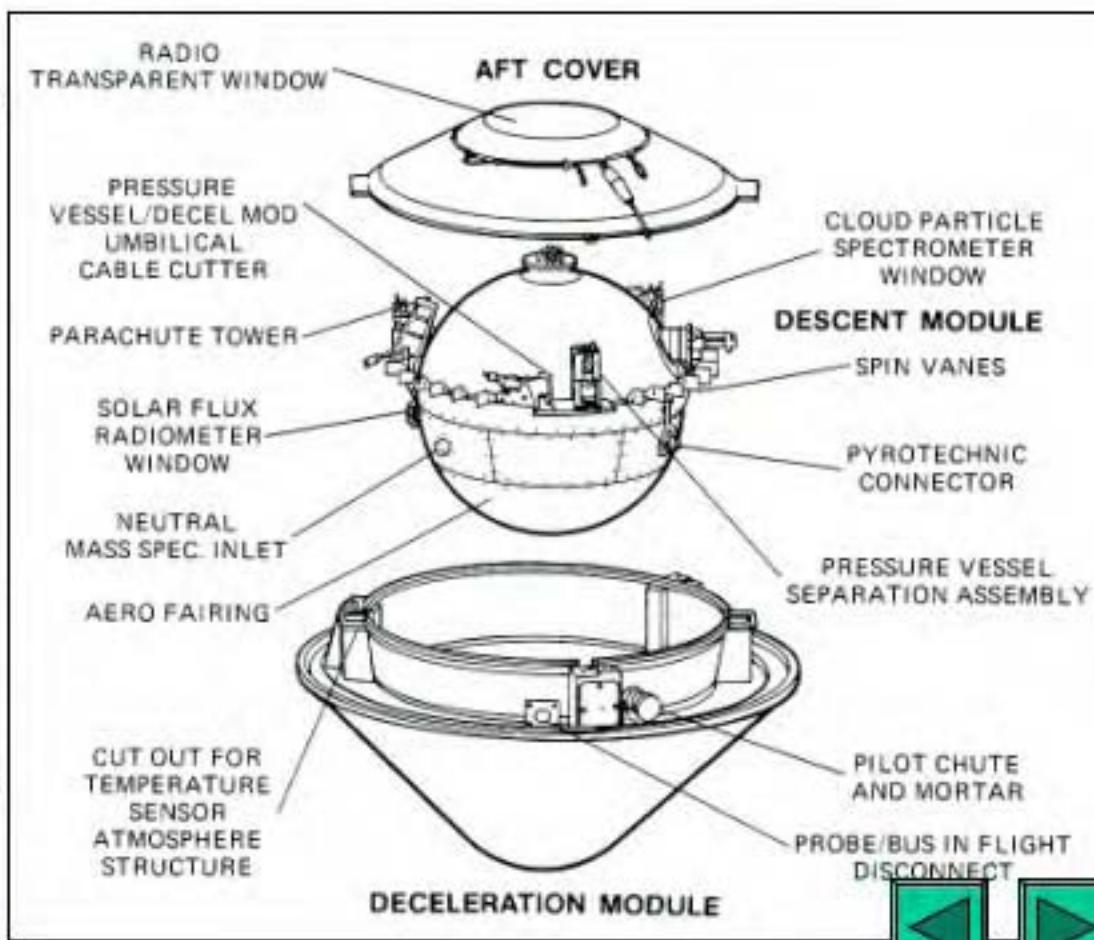
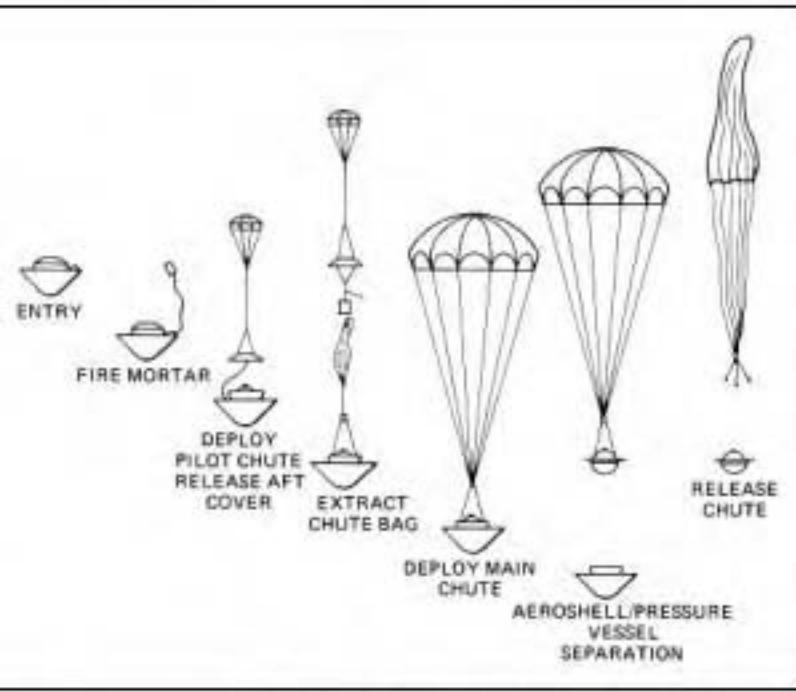


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Pioneer Venus Large Probe

- 315 kg, 1.5 m diameter, 7 experiments
- Gas Chromatograph, PI Vance I. Oyama
- Neutral Particle Mass Spectrometer, PI John D. Hoffman



Fimmel, R., Colin, L., Burgess, E.,
Pioneer Venus, NASA SP-461, 1983.

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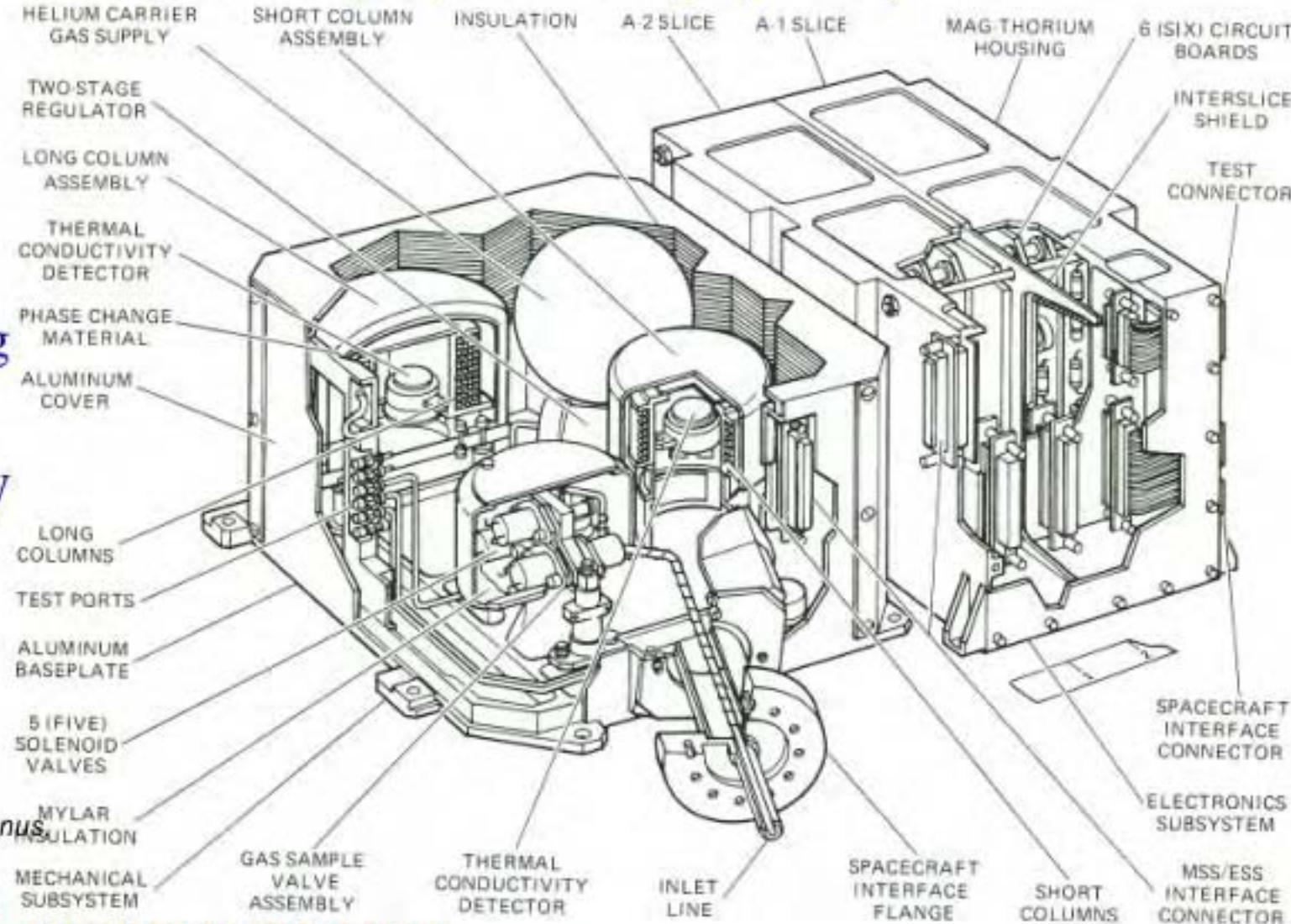
Large Probe Gas Chromatograph (LGC)

- 2 GC columns in parallel: poropak N and polydivinyl benzene polymer

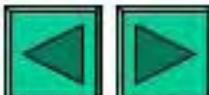
- Thermal conductivity detectors

- Mass: 3 kg

- Average Power: 4.6 W



Fimmel, R., Colin, L.,
Burgess, E., *Pioneer Venus*,
NASA SP-461, 1983.



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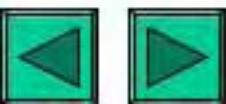
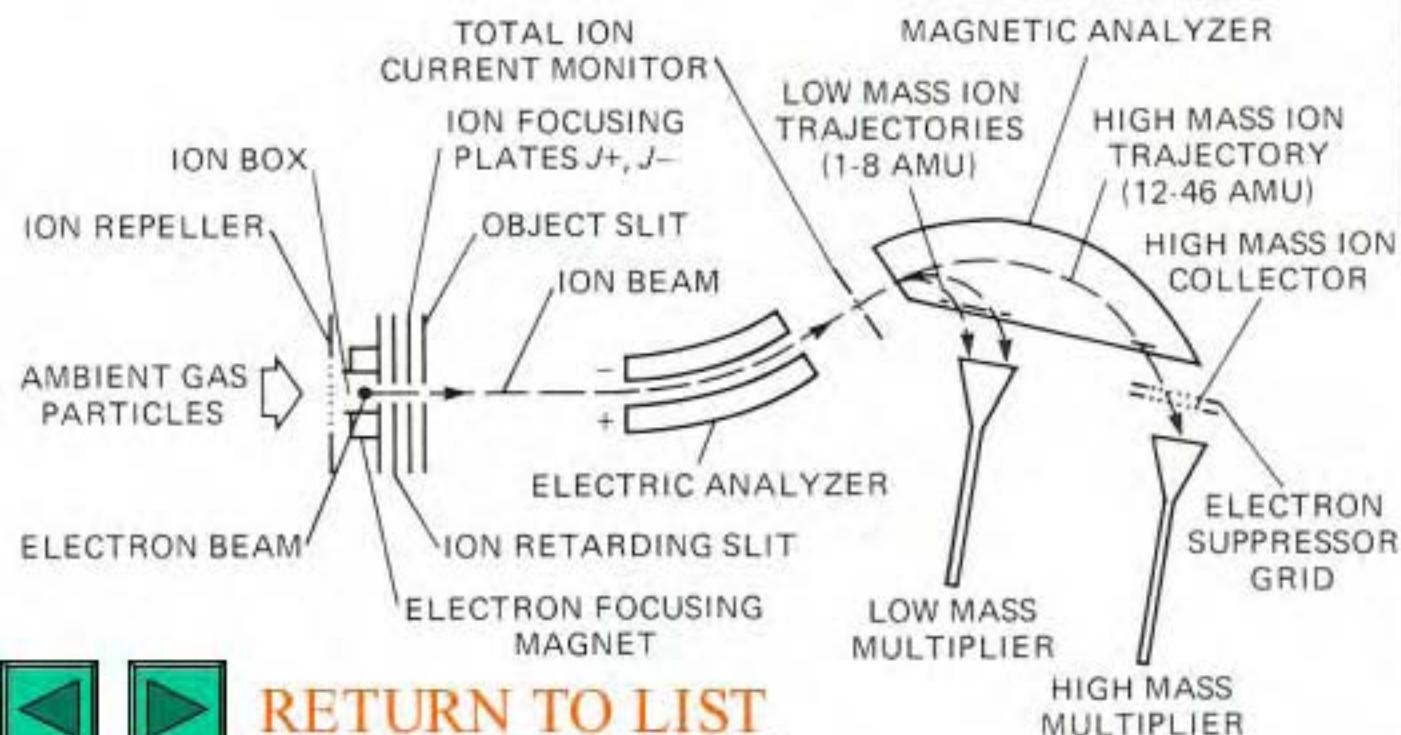
Large Probe Neutral Particle Mass Spectrometer (LNMS)

- Double-focusing magnetic deflection mass spectrometer
- Ceramic micro-leak gas inlet
- Mass range: 1 to 212 u
- Dynamic range: 1×10^7
- Mass: 9 kg
- Average Power: 12 W



Bus Neutral Mass Spectrometer (BNMS)

- Magnetic deflection, double-focusing mass spectrometer
- Mass range 1 to 46 u
- Mass: 5 kg
- Average Power: 6 W



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Fimmel, R., Colin, L.,
Burgess, E., *Pioneer
Venus*, NASA SP-461, 1983.

Combined Results of the Pioneer Mass Spectrometers and GC Lower Atmosphere Data

Gas	Amount, ppm
Argon	40–120
40/36	1.03–1.19
38/36	0.18
Carbon dioxide	96%
Carbon monoxide	20–28
Krypton	0.05–0.5
Neon	4.3–15
Nitrogen (percentages)	3.41% (at 24 km) ^a ; 4% ^b 3.54% (at 44 km) ^a 4.60% (at 54 km) ^a
Oxygen	16 (at 44 km) ^a ; <30 ^b 43 (at 55 km) ^a
Sulfur dioxide	185 (at 24 km) <10 (at 55 km)
Water	20 (at surface) 60–1350 (at 24 km) 150–5200 (at 44 km) 200–<600 (at 54 km)

^aLGC

^bLNMS

After J. H. Hoffman; based on six different instruments — four mass spectrometers and two gas chromatographs.

Fimmel, R., Colin, L.,
Burgess, E., *Pioneer Venus*, NASA SP-461,
1983.



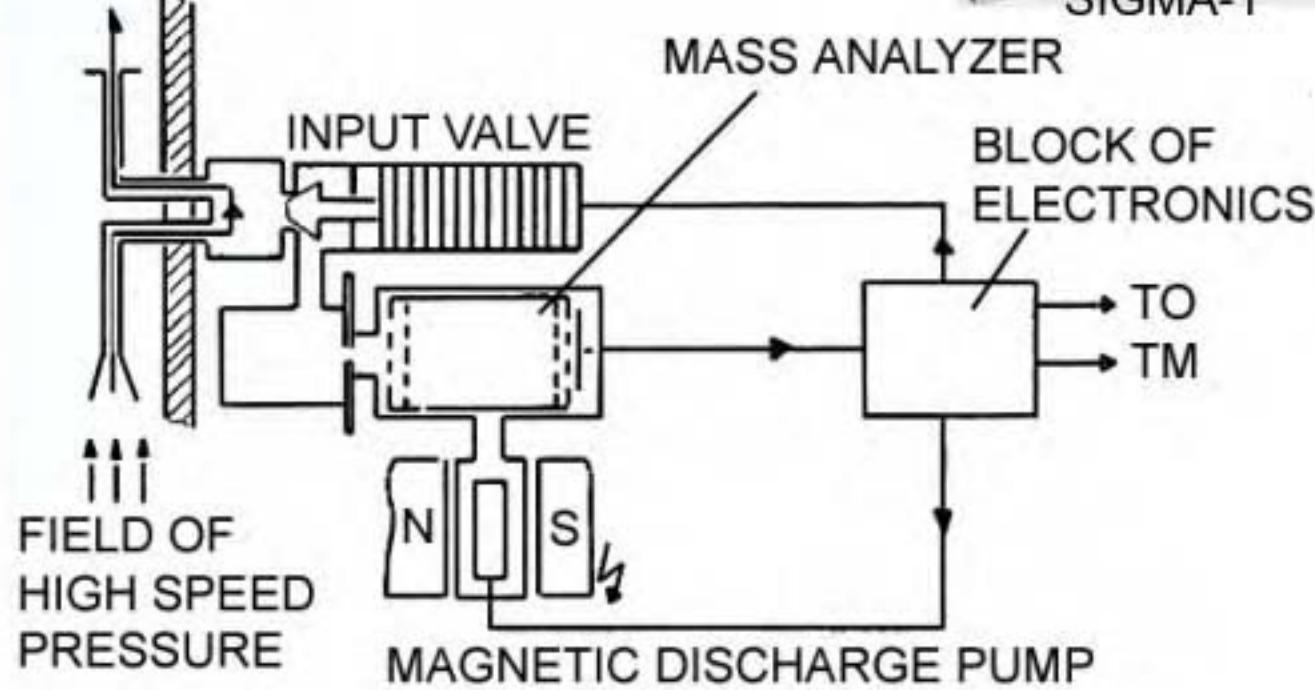
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Soviet Venera-11 and 12 at Venus

- Launch: September 9, 1978 (V-11); September 14, 1978 (V-12)
- Entry and Landing: December 25, 1978 (V-11);
December 21, 1978 (V-12)
- Bennett type radio-frequency mass spectrometer
- Mass range 11-105 u
 - Operated from 23 km to surface
- SIGMA-1 GC experiments



SIGMA-1



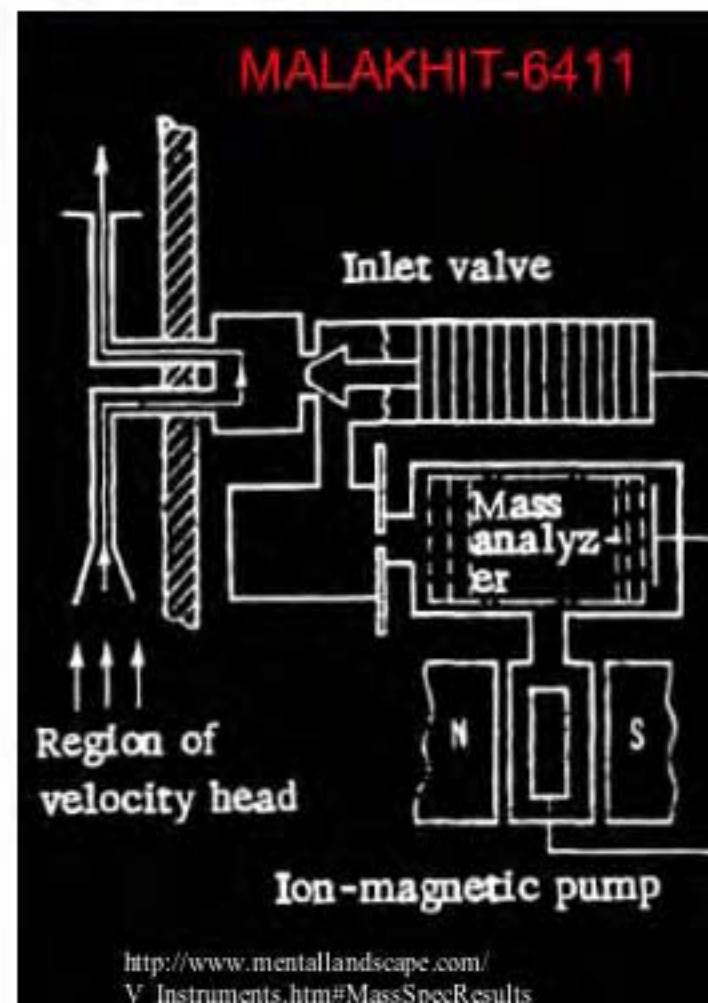
Ref: NASA TM-75455 1979, Mass Spectrometry Measurements of the Composition of the Lower Atmosphere of Venus, Istomin, V.G. et al.

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Soviet Venera-13 and 14 at Venus

- Launch: October 30, 1981 (V-13); November 4, 1981 (V-14)
- Entry and landing: March 1, 1982 (V-13); March 5, 1982 (V-14)
- MALAKHIT-6411 mass spectrometer
 - Operated from 26 km to surface
 - Mass range: 11-138 u
 - 9.5 kg mass, 17 W average power
 - Sensitivity limit 50 ppb
- SIGMA 2 GC experiment
 - Operated from 58 km to surface
 - 3 columns
 - 2 neon ionization detectors,
1 electron-capture detector



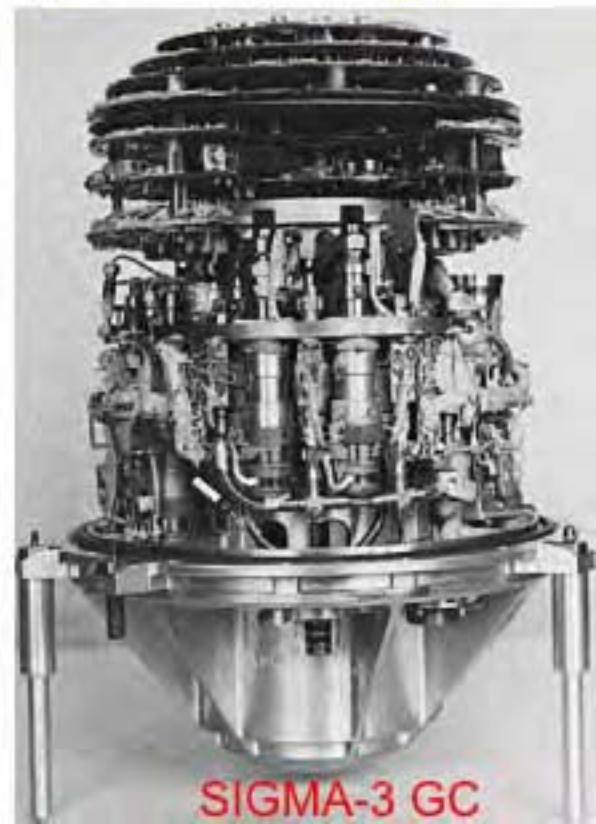
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Soviet Vega 1 and 2 Venus Landers at Venus



Vega mass spectrometer

- Launch: Vega-1 on December 15, 1984; Vega-2 on December 21, 1984
- Entry and Landing: Vega-1 on June 11, 1985; Vega 2 on June 15, 1985
- Parachutes and aerodynamic braking



SIGMA-3 GC

- 3-dimensional quadrupole ion trap MALAKHIT-M mass spectrometer with pyrolyzer
- SIGMA-3 gas chromatography experiment

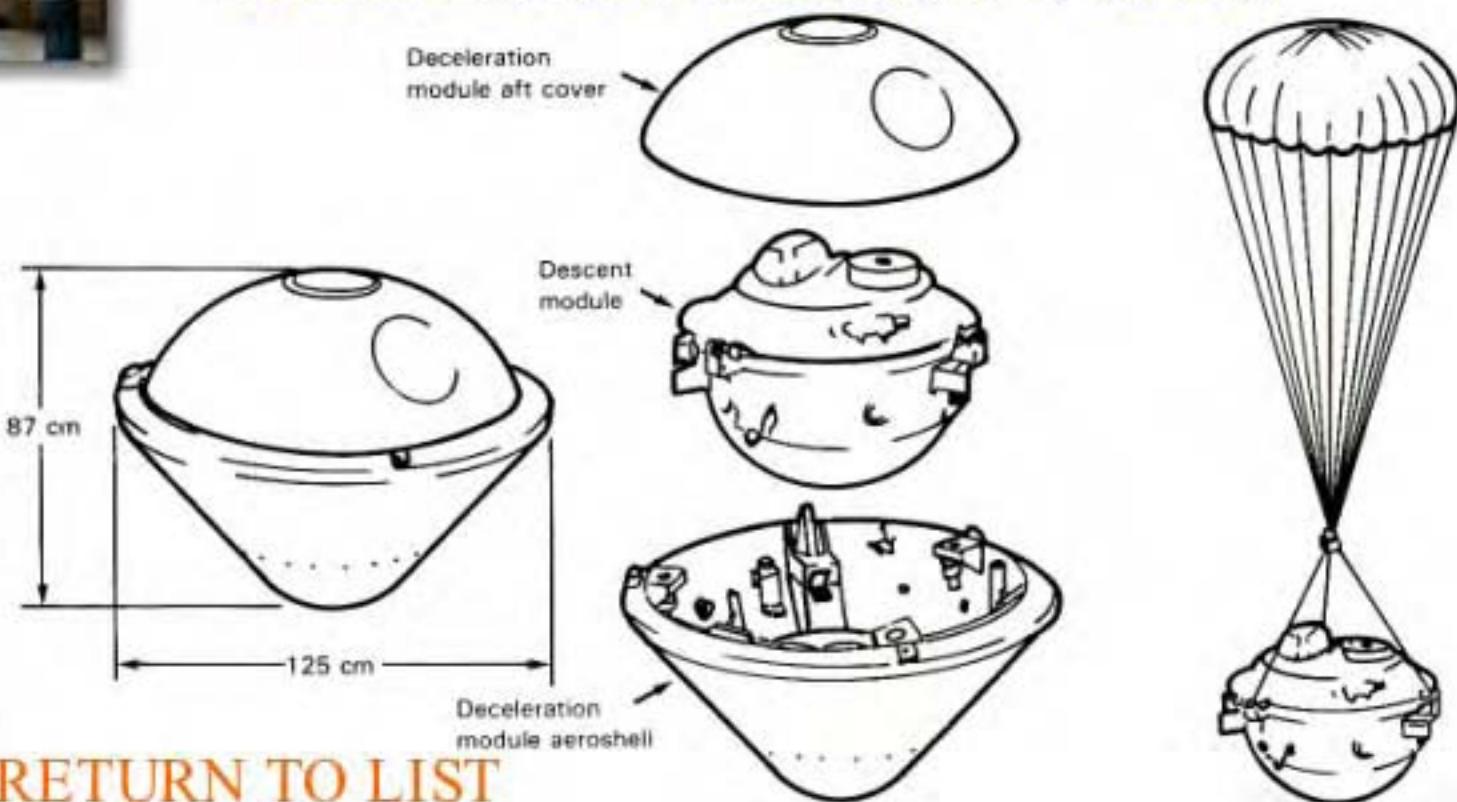
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Galileo Probe at Jupiter

- Launch: October 18, 1989
- Entry: December 7, 1995
- Probe mass: 340 kg
- 5 experiments
- Descent lasted 58 minutes (0.4 to 23 bar)



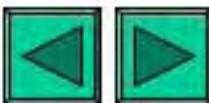
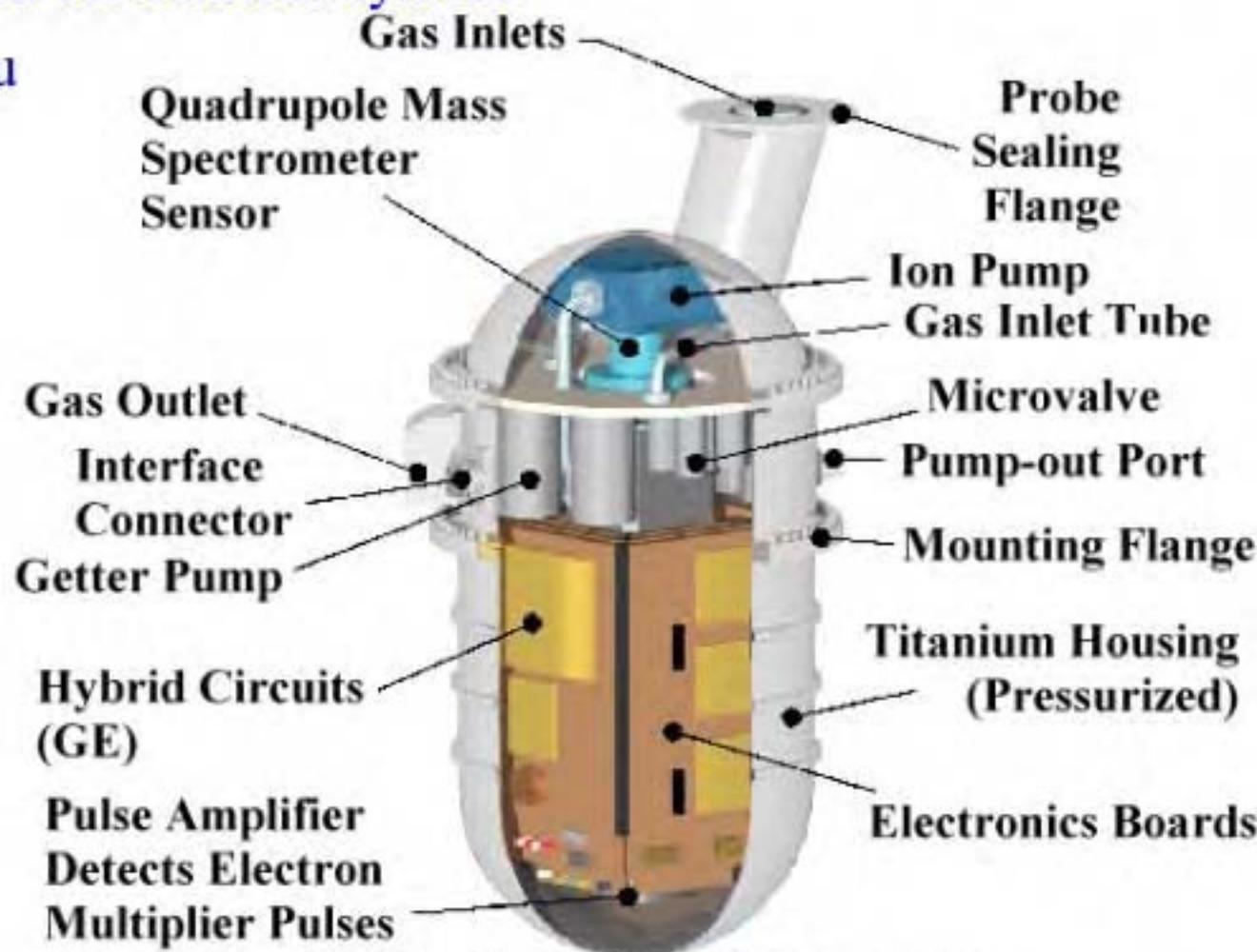
Yeates, C., Johnson, T.,
Colin, L., Fanale, F.,
Frank, L., Hunten, D.,
*Galileo: Exploration of
Jupiter's System*, NASA
SP-479, 1985.



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Galileo Probe Mass Spectrometer (GPMS)

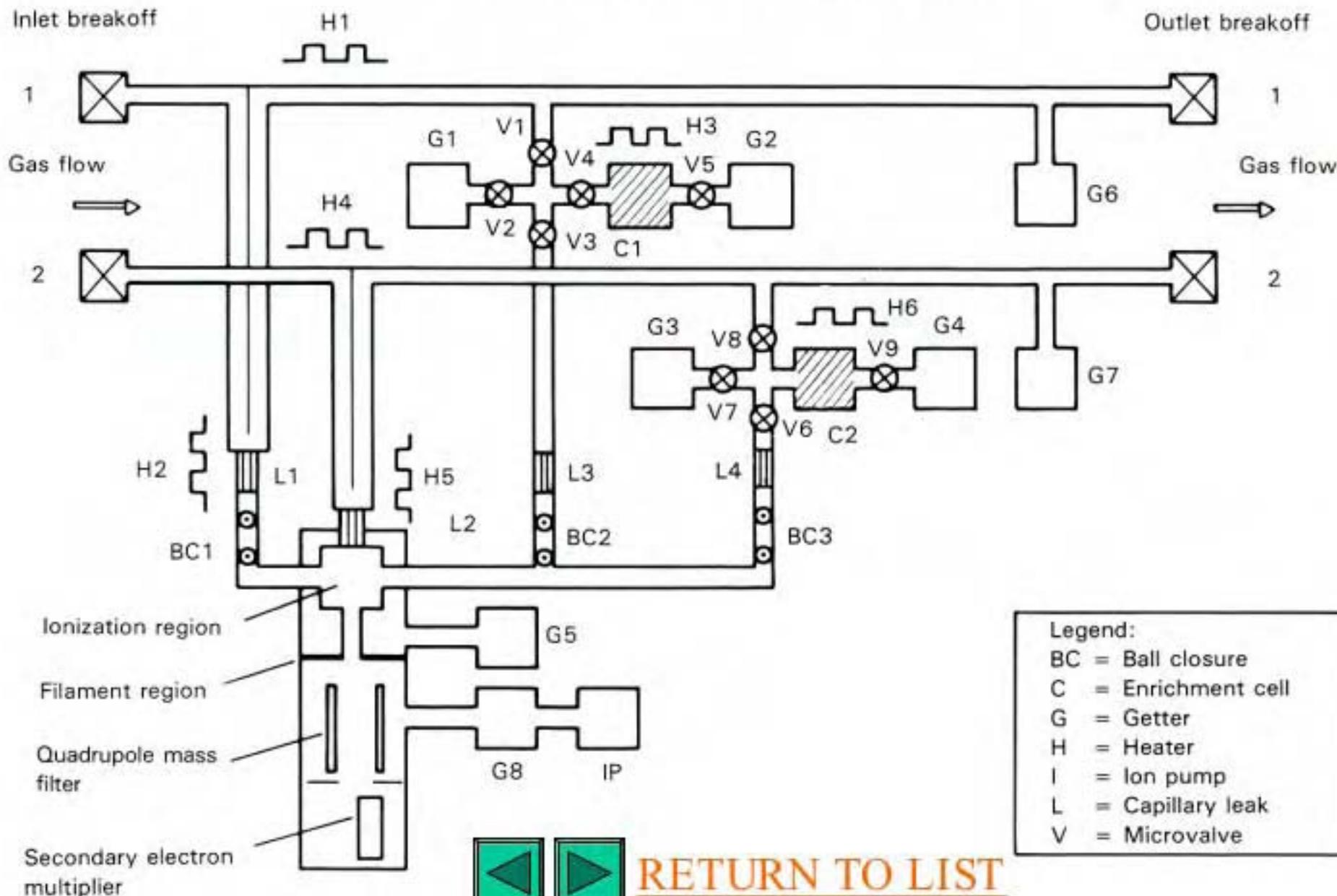
- Quadrupole sensor with 2 electron impact filaments
- Sample trapping and enrichment system
- Mass range 1-150 u
- Mass: 13.2 kg
- Average Power:
22 W



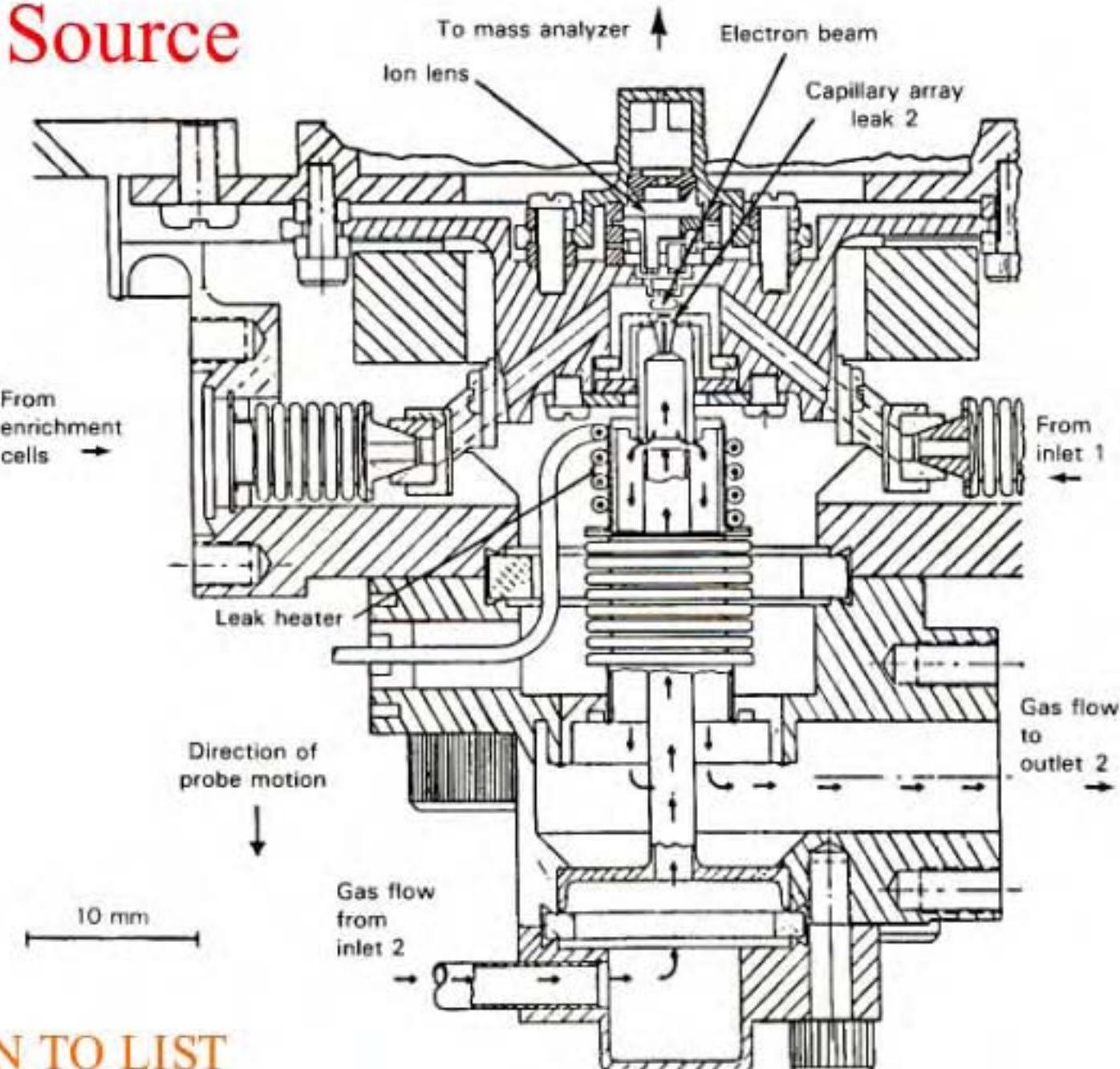
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Galileo Probe Mass Spectrometer
Configuration Overview

GPMS Inlet System



GPMS Ion Source



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Cassini- Huygens Probe at Titan

- Heat shield and parachutes
- 6 instruments
- Probe and instruments survived impact and acquired data for over 1 hour on the surface

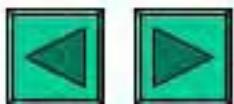


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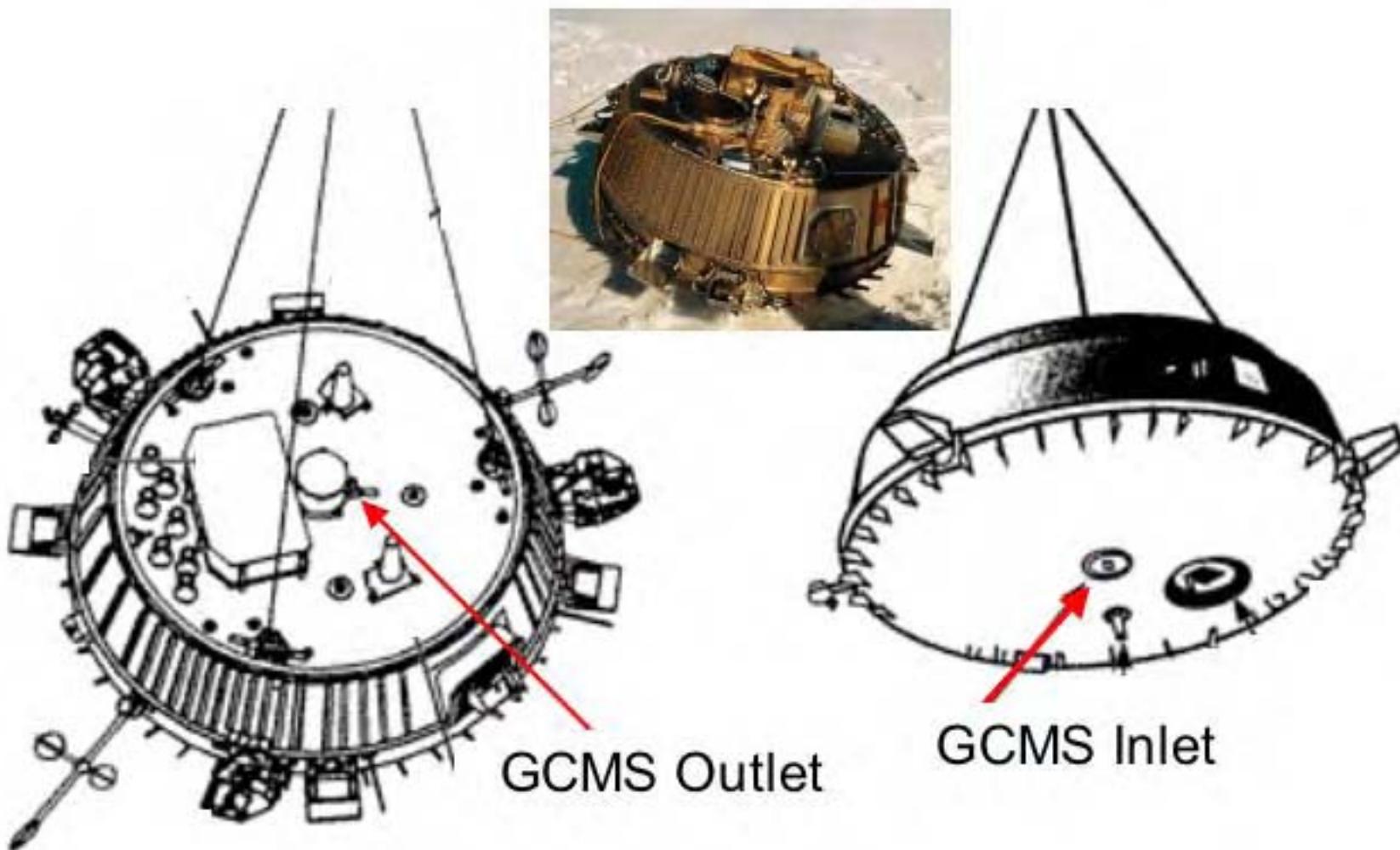
Huygens Gas Chromatograph Mass Spectrometer (GCMS)

- Quadrupole mass spectrometer with a secondary electron multiplier detection system, 5 electron impact ion source, 3 gas chromatographic columns
- Sample trapping and enrichment system
- 17.3 kg mass, 41 W average power
- Mass range 2-141 u
- Dynamic range $\geq 1 \times 10^8$
- Resolution 1×10^{-6} for adjacent half masses up to 60 u, less for higher masses
- Sensitivity limit 1×10^{14} counts/s/hPa source pressure



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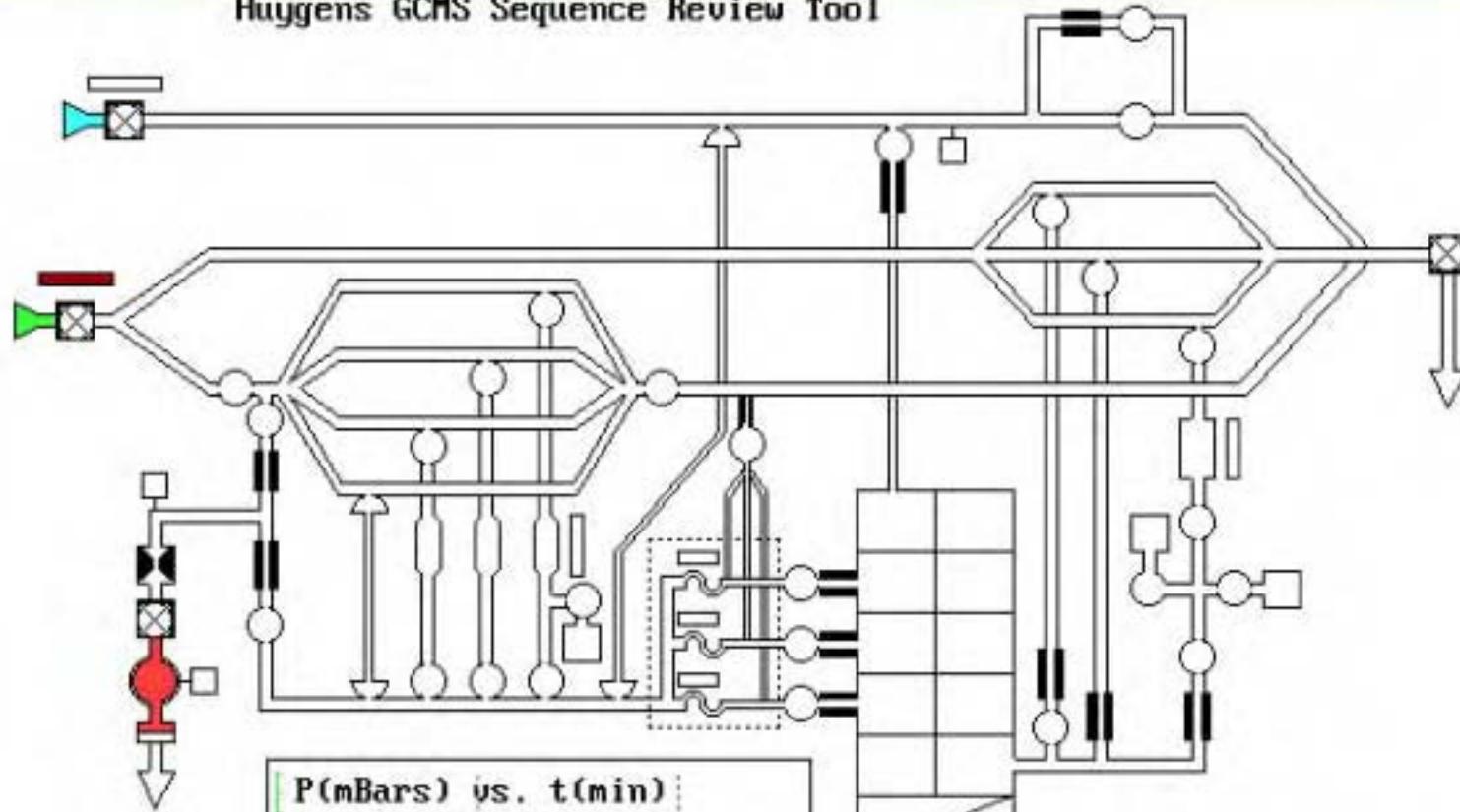
GCMS Inlet Location



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GCMS

Huygens GCMS Sequence Review Tool



P(mBars) vs. t(min)
Nominal Entry

60

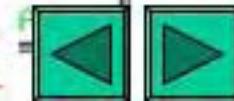
1000
<Tab>

Esc Quit
Fn Mode
1

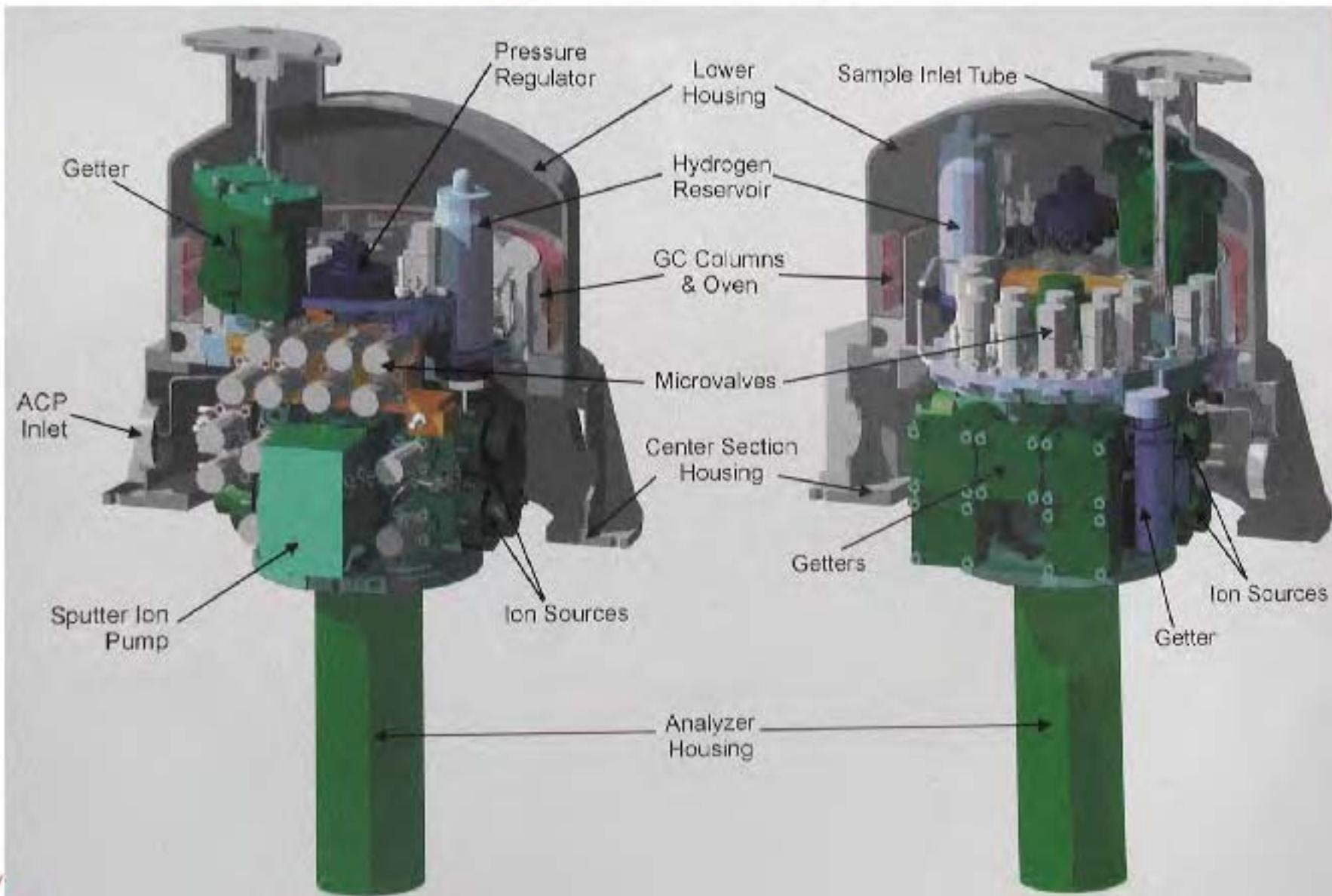
Manual Mode
Ring Key

Time: 0:00:00
Op: HI ON

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GCMS Flight Configuration



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